WATER EXTRACTABLE DOC, DON AND NUTRIENTS IN URBAN SOILS:

EFFECTS OF SODIUM SOURCE AND EXPOSURE INTERVAL

A Thesis

by

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ABSTRACT

Dissolved organic carbon (DOC) and organic nitrogen (DON) in surface waters represent a loss of sequestered C and N from terrestrial to aquatic ecosystems (allochthonous inputs). Recently, aquatic DOC and DON concentrations have been increasing. Multiple studies have suggested that increased sodium inputs in urban centers contribute to this increasing DOC and DON. To determine the effect of sodium exposure to urban soils, this study took samples from 3 different cities exposed to three different sodium types: Bryan-College Station, TX (sodic irrigation water), Frederick, MD (deicing salts), and Galveston, TX (sea salt deposition). Multiple regression models found that the best independent variables for predicting DOC were sodium type, pH, EC, ammonium-N, phosphate-P, DON, magnesium, boron, iron, zinc, and manganese. The best independent variables for DON were time of sodium exposure, sodium type, nitrate-N, phosphate-P, magnesium, sodium, zinc, manganese, and DOC. The difference in independent variables for estimating water extractable DOC and DON concentrations in urban soils suggests that the mechanisms controlling DOC and DON desorption are extremely complex. It is likely that the mechanisms controlling DOC are directly or indirectly related to pH while the mechanisms controlling DON are controlled indirectly by pH as well as microbial respiration rates.

The second part of this study aimed to look at the effect of 4- and 2- hour shakes on measured concentrations of DOC, DON, TDN, NH_4, NO_3, and PO_4. As there is no definitive extraction method for DOC and DON, cross comparison across studies

is unreliable. Using different methods can result in overestimation through nutrient release through microbial release during extraction or underestimation through organic nutrient degradation. To look at the impact of shake time on measured analyte concentrations, results of 2 and 4 hour shakes were compared across 4 cities: Chicago, IL, Frederick, MD, Galveston, TX, and Bryan-College Station, TX. The results of 2 and 4 hour shakes for DOC, DON, or nutrients (except NO_3) were highly variable across cities, suggesting that each city's distinct soil properties were responsible for the differences in analyte response to shake time.

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TABLE OF CONTENTS

Page

LIST OF FIGURES

Page

LIST OF TABLES

Page

CHAPTER I

INTRODUCTION AND LITERATURE REVIEW

1.1 Urbanization and land use change

Urbanization has been increasing dramatically in recent years. In 2014, the global urban population rose to 3.9 billion, compared to 746 million in 1950 (United Nations Department of Economic and Social Affairs Population Division, 2014). In North America alone, 82% of the population lives in urban centers (United Nations Department of Economic and Social Affairs Population Division, 2014). With urbanization comes disturbance of natural ecosystems. In addition to the more widely recognized ecosystem disruptions such as deforestation and wetland dredging, natural soil processes are also being disturbed. Urbanization and its land management has been linked to changing soil chemistry which can have implications on surface water chemistry (Aitkenhead-Peterson et al., 2010; Aitkenhead-Peterson et al., 2009; Green et al., 2008a; Green et al., 2008b; Steele and Aitkenhead-Peterson, 2012b; Steele et al., 2010).

Alkalinization of streams has been increasing due to the weathering of geologic formations, concrete, pavement and other urban structures due to acid rain (Kaushal et al., 2013). Alkalinity is the a function of inorganic and organic acids and bases (typically bicarbonates and carbonates) and results in alkaline pH (Kaushal et al., 2013). When concrete and limestone are weathered, it causes the release of H^+ ions, ultimately, resulting in the dissolution of bicarbonates and carbonates, which are responsible for

increasing alkalinity and calcium concentrations (Kaushal et al., 2013; Stets et al., 2014). Increased alkalinity is essentially a buffer and prevents acidification of watersheds; however, it can have detrimental environmental consequences. Alkalinity can have significant impacts on the carbon cycle (Kaushal et al., 2013; Li et al., 2008). Generally, silicate weathering of rock sequesters CO_2 and converts it into dissolved inorganic carbon (DIC); however, weathering of rock by other acids (sulfuric, nitric) can cause CO_2 production due to their acidity. Therefore, increased weathering of rock due to acid deposition may cause a net increase in atmospheric CO_2. While acid rain has substantially decreased since the 1970's, the concentration of acids deposited due to rainfall is still high enough to cause the weathering of urban structures.

Increased sodium and chloride in urban watershed has also been linked to increasing urbanization due to increased deicing salt use, WWTP effluent, and highly sodic irrigation water (Steele and Aitkenhead-Peterson, 2012b; Steele et al., 2010). High concentrations of these anions can be toxic to fish, invertebrates and plants (Steele et al., 2010). For urban landscapes irrigated with highly sodic irrigation water ($NaHCO_3$), sodium adsorption ratio (SAR) and alkalinity of the irrigation water were shown to be the best predictors of water extractable (WE) dissolved organic carbon (DOC) and dissolved organic nitrogen (DON) (Steele and Aitkenhead-Peterson, 2012b). Other studies have shown increased DOC concentrations in solution in response to long-term deicing salt exposure (Green et al., 2008b). In essence, increased sodicity and salinity are observed in urban watersheds as a response to increasing urbanization.

1.2 Sources, transport, fates and the importance of dissolved organic carbon and dissolved organic nitrogen

DOC and DON are components of DOM. DOC is defined as material composed of small organic molecules and highly polymeric humic substances that pass through a 0.45 µm filter (Thurman, 1985). DON is comprised of amino acids, proteins and peptides (Carrillo-Gonzalez et al., 2013). DON and DOC are influenced by each other and are typically tightly coupled in undisturbed ecosystems (McDowell, 2003). DOC and DON in surface waters represents a loss of sequestered C and N from terrestrial ecosystems (allochthonous inputs) and once in surface waters they are implicated in the formation of trihalomethanes when surface water is used for municipal water supplies (Fujii, 1998; Gough et al., 2014). Recent research has found that DOC concentrations and exports from urban watersheds have shown large increases relative to their native land uses (Aitkenhead-Peterson and Steele, 2016; Aitkenhead-Peterson et al., 2009; Petrone, 2010). While DON concentrations and exports are also increased, their relative proportion of total N has significantly decreased (Aitkenhead-Peterson and Steele, 2016; Aitkenhead-Peterson et al., 2009). Inorganic nutrients NH_4-N, NO_3-N and PO_4-P are also observed in high concentrations in urban watersheds, enhanced by the loss of riparian buffer area (Allan, 2004).

Allochthonous sources of DOC to aquatic ecosystems were described by Aitkenhead-Peterson et al. (2003) and suggest that the primary source of DOC is from the organic horizons of soil, with contributions from mineral soil, leaf litter, root death and decay, rainfall and throughfall. However, concentrations of DOC vary significantly

in a watershed soil (Table 1). Concentrations are significantly lower once soil solution reaches mineral soil horizons, due to the adsorption of DOC to soil minerals (Jardine et al., 1989; Kaiser et al., 1996), soil solution ionic strength (Evans et al., 1988; Schulthess and Huang, 1991) and mineralization of DOC by the soil microbial community (Cioce and Aitkenhead-Peterson, 2015; McDowell et al., 2006; Yano et al., 1998). DOC from any of the aforementioned DOC pools can make its way to aquatic ecosystems. In essence, the fate of DOC is adsorption, mineralization, migration to groundwater, or runoff into aquatic ecosystems.

Table 1: Concentrations of DOC observed in multiple studies.

Location	Soil Classification	Source	DOC Concentration (mg L^{-1})	Reference
Throughfall				
Oregon, USA	Inceptisol, Spodosol, Ultisol	Spruce-Fir forest, throughfall	5.5	(Bockheim and Langley Turnbaugh, 1997)
Schefferville, Canada	Inceptisol and Spodosol	Fen catchment, throughfall	48.5	(Koprivnjak and Moore, 1992)
Organic soil				
Bavaria, Germany	Dystrochrept and Haplorthod	Spruce forest with acidic soils	36.0	(Guggenberger and Zech, 1993)
Iowa, Wisconsin	Mollisol	Prairie, Organic soil horizon	4.3	(Quideau and Bockheim, 1997)
Location	**Soil Classification**	**Source**	**DOC Concentration (mg L^{-1})**	**Reference**
Mineral soil				
Wales, UK	Gley	Ephemeral forest	6.3	(Reynolds and Hughes, 1989)
Lake Calado, Amazonas, Brazil	Oxisol	Tropical forest, mineral soil horizon	1.9	(Williams et al., 1997)
Reefton, New Zealand	Spodosol	Deciduous forest, mineral soil horizon	12.0	(Moore and Jackson, 1989)
BCS, TX	Alfisol	Urban	20.4-52.5	(Aitkenhead-Peterson et al., 2009)

Allochthonous sources of DON are similar to DOC and include contributions from mineral soil, leaf litter, root death and decay, rainfall and throughfall with organic horizons being the primary source (Aitkenhead-Peterson et al., 2003). DON concentrations decrease in mineral soils due to its increased ability to adsorp dissolved organic matter (DOM). Conversely, DON concentrations are higher in organic soils; however, organic horizons may be a higher source of DOC than DON.

1.3 Ecological significance of DON and DOC

Increased allochthonous DOM, specifically DOC, includes the provision of substrate for microbial production in both terrestrial and aquatic systems (Cioce and Aitkenhead-Peterson, 2015; McDowell et al., 2004). As one of smallest units of the food chain they are vital components of 2 ecosystem functions which include a food source for higher trophic levels and mineralization of organic detritus that regenerates ecosystem nutrient concentration (Harbott and Grace, 2005). As the microbial community is a lower trophic level, it is an important regulator of ecosystem health. It can, therefore, be said that DOM may be a driver in healthy food web and ecosystem functioning. DOM also plays a role in the attenuation of ultraviolet (UV-B) radiation which, in turn, provides protection for many aquatic species (Williamson and Zagarese, 1994). Addition of sodium from urban environments is hypothesized to indirectly change the composition of DOM in the environment (Green et al., 2008a; Green et al., 2008b; Steele and Aitkenhead-Peterson, 2011; Steele and Aitkenhead-Peterson, 2012a; Steele and Aitkenhead-Peterson, 2012b). As sodium increases in the environment it

replaces monovalent cations such as H+ on soil exchange sites, which will increase the soil pH and, consequently, increase the solubility of the humic acid fraction of DOM (Green et al., 2008b).

Sodium may also increase DOC and DON loss by maintaining the electroneutrality of solution. For example, sodium eventually replaces divalent cations on soil exchange sites. To counteract this, an equivalent mass of anions must be removed from anion exchange sites in order to maintain electroneutrality. It also causes increased desorption of cations, such as H^+, which increases soil pH and DOC and DON solubility. In addition, long term exposure to sodium results in disaggregation of soil and dispersion of clay particles, reducing small soil pores, ultimately, increasing the bioavailability of DOM to microbes (Baldock and Skjemstad, 2000; Steele and Aitkenhead-Peterson, 2012b). However, urban soils have been shown to decrease microbial biomass by 50% compared to rural soils due to lower quality OM; however, researchers were unclear as to the reason behind litter quality variability (Carreiro et al., 1999). An increase in DOC would, presumably, provide a substrate for microbes and allow for the growth of microbial communities. It has been shown to increase soil microbial biomass, but actually reduces microbial respiration rates (Wong et al., 2008). Meaning the microbial communities are larger, but less active. Decreased metabolization of desorped DOM by microbial communities may result in increased concentrations of allochthonous DOC and DON transported to urban streams, which could potentially alter stream ecosystem functioning. While DOM is an important microbial substrate, changes in its

concentration or molecular structure from simple carbon chains to aromatic C and complexed metal-DOM, may negatively impact watershed soil and stream ecosystems.

Negative effects of increased allochthonous DOC include the mobility of metals (Driscoll et al., 1988; Martell et al., 1988). A study conducted at the Hubbard Brook Experimental Forest found that hillslope influenced metal concentrations in stream water. Lower elevations had shallower soil and higher input of organic solutes and stronger acidic solutions which caused inflated concentrations of trace metals (Pb and Fe) in streams (Driscoll et al., 1988). Therefore, lower concentrations of DOC led to higher retention of metals in soil. Conversely, increased stream DOC inputs resulted in metal desorption from the soil and increased loading into the aquatic environment.

DOC has been shown to reduce adsorption of pesticides to soils (Worrall et al., 1997a; Worrall et al., 1997b). Worrall et al. (1997a) recognized the role DOC played in the adsorption of the pesticide isoproturon. Initially, when soil was dry, pesticide adsorption increased. As the soil was wetted and soluble organic matter released, DOC concentrations increased while pesticide sorption decreased. Researchers attributed increased pesticide concentrations in solution to two potential mechanisms: (1) the pesticide exchanged to an adsorption site on DOC instead of insoluble organic matter or (2) the isoproturon initially adsorped to the DOC and when the DOC was released due to soil wetting, the pesticide was released into solution. The researchers did not find any DOC-isoproturon complexes and were unable to observe these complexes possibly due to the bonding weakness. Worrall et al. (1997a) mentioned atrazine-DOC complexes were weak and dissociated readily. A study by Worrall et al. (1997b) confirmed the

findings of Worrall et al (1997b) on isoprotural-DOC complexes. Ultimately, increased DOC loss from watershed soils could result in increased pesticide runoff into streams.

While DOM is an important substrate for microbial growth, it also provides idyllic conditions for the regrowth of *E. coli* (Bolster et al., 2005; McCrary et al., 2013). Recent work has shown that *E. coli* can persist and grow outside of their host in both soil and water, which had previously been thought impossible (Bolster et al., 2005; McCrary et al., 2013; Vital et al., 2008). McCrary et al. (2013) found that when treated WWTP effluent was incubated in stock solutions of vegetation (grass or herbaceous leaves), *E. coli* populations increased. Also observed in these experiments was the decline in concentrations of DOC and, more notably, DON which led researchers to conclude that DON was an important substrate for *E. coli* growth. Vital et al. (2008) also stated that carbon concentrations do not have to be high in order to promote regrowth; concentrations typically found in natural environments are high enough for regrowth to occur. Though increases in DOM concentrations in the aquatic environment may further promote *E. coli* growth in urban streams, specifically those downstream from WWTPs.

DOC has also been linked to carcinogenic trihalomethane (THM) production in chlorinated drinking water (Stevens et al., 1976). When water containing humic acids was treated at drinking water treatment plants and came into contact with chlorine, it resulted in the presence of trihalomethanes. THMs were formed at all pH levels, though higher concentrations were formed at high pH which is likely due to humic acid being more soluble at higher pH. At a low pH of 6.7, only 0.7% of total humic acids underwent

a reaction to form THMs while at pH 9.2, 1.4% of all DOC underwent a reaction to form THMs (Stevens et al., 1976).

1.4 DOC:DON ratio

DON tends to be related to DOC within ecosystems (Aitkenhead-Peterson et al., 2005). These ratios vary naturally with respect to environmental changes, and decrease in response to increased precipitation and stormflow (Bernal et al., 2005). This change was attributed to an instream source of C and terrestrial origin of N. During periods of rain, water flow paths would travel through OM with high N content and result in lower DOC:DON ratios in streams (Bernal et al., 2005). In urban systems, there tends to be lower DOC:DON ratios due to anthropogenic loading of N from agriculture and dense urban centers (Pellerin et al., 2006); however, there is also a decrease in DON:TDN ratio a higher proportion of total N becoming nitrate (Stanley and Maxted, 2008). The increase in DON and nitrate were attributed to N input in excess of biological demand (Stanley and Maxted, 2008). Understanding the relationship between them will be helpful in determining total losses of C and N from terrestrial ecosystems. As they are related, the observed increase in DOC due to sodium exposure will also result in loses of DON, ultimately altering carbon and nitrogen cycles. In situations where there is a high input of inorganic-N as fertilizer the DOC and DON dynamics may be decoupled (McDowell et al., 2004).

Hydrologic flow in forested systems has been shown to be an important determinant in DOC:DON ratio. Soil C:N ratio was ascertained to explain 75% and

73% of the variability in DOC and DON, respectively, in a forested watershed in Nova Scotia (Aitkenhead-Peterson et al., 2005). This suggests that the DOC:DON ratio is influenced by proximity of organic layers to hydrological flow paths (Aitkenhead-Peterson et al., 2005). During times of higher rain fall or snowmelt, stream flow is higher and has increased contact with soil within the organic horizon. Increased runoff during these periods also results in increased contact with the organic topsoil layer of forest floors, and increased DOC loading into streams (Aitkenhead-Peterson et al., 2005). This trend was seen both seasonally and annually, but was more pronounced during annual timescales (Aitkenhead-Peterson et al., 2005).

Lepisto et al. (2013) observed similar trends within forested watersheds in Finland. Seasonal variation in DOC:DON ratio was attributed to changing average rainfall. During the dormant season ratios increased to 30-35, while during the summer they decreased to 27-30. Researchers attributed this trend to increased precipitation but also more effective DON production during the summer. Essentially, Lepisto et al. (2013) observed similar trends to those determined by Aitkenhead-Peterson et al. (2005). During wetter seasons, the ratio between DOC and DON was much lower due to increased hydrologic flow and residence time with organic soil layers.

Studies quantifying variability in DOC:DON and watershed soil C:N ratios in urban soils are minimal. Researchers have found that in urban soils C:N ratios are 13-14% higher in particulate organic matter (POM) fractions compared to rural soils (Scharenbroch et al., 2005). They attributed this change to the change in composition of the soil microbial community. Instead of a large quantity of nitrifying organisms, the

microbe community was dominated by “general purpose decay organisms.” This changes the ratio of nitrogen in the soil relative to carbon. Studies conducted in forested watersheds have shown DOC:DON variance can be explained by the ratio of C:N. Studies denoting this variance in urban soils are scarce. If the C:N impact on DOC:DON holds true, it can be hypothesized that the DOC:DON in urban soils may mirror that found in the study conducted by Scharenbroch et al. (2005).

1.5 Nutrients in urban soils

1.5.1 Phosphate

Phosphorus is generally the limiting nutrient in many ecosystems and is typically the limiting factor in plant productivity (Tye et al., 2016). Sources of excess phosphorus in urban streams and soils is typically WWTP effluent and phosphate fertilizer runoff (Tye et al., 2016). Excess phosphorus can enhance primary productivity and causes an overgrowth of algae, ultimately depleting dissolved oxygen in water ecosystems. Knowing additional mechanisms leading to potential release from soils is important in order to prevent further phosphate pollution.

In a natural Fen ecosystem with fertilization runoff inputs, phosphate release increases during long term flooding events (Beltman et al., 2014). The effects of these events on phosphorus were only seen over long periods of time, as the process of phosphate release is not rapid. The researchers hypothesized this was potentially due to 3 factors: (1) slow microbial activity, (2) slow infiltration of water into soil or (3) a delay in anoxic conditions (Beltman et al., 2014). Beltman et al. (2014), conducted their study

over a period of 216 days, but another study conducted phosphate release over a 15 year period in sulfate rich water and observed similar trends *(*Zak et al., 2009*)*. Researchers hypothesized that sulfate was either replacing loosely bound phosphate in soil or that Fe (III) hydroxides released bound phosphate when sulfate reduction occurred. In essence, in natural ecosystems with fertilizer runoff influence, phosphate mobilization will occur under long-term flood conditions. It occurred in natural Fen ecosystems with and without major sulfate influence, but both ecosystems were influenced by phosphate fertilizers.

There have been few studies on the relationship between PO_4-P and DOC. Though researchers did find that in acidic soils (pH~5.5) in the presence of DOC, about 14.6-19.9% less phosphate was retained in the soil (Fernandez-Perez et al., 2005). Researchers attributed this to the decrease in solution pH when DOC was added. The soil used in this study was calcareous. In calcareous soils, phosphate tends to precipitate as calcium phosphate which has a neutral charge. (Fernandez-Perez et al., 2005). DOC is negatively charged (Carrillo-Gonzalez et al., 2013) and would adsorp more strongly to soil than calcium phosphate, causing increased mobilization of calcium phosphate from soil. This trend may however not be typical in every soil type. More research needs to be conducted in different soil types to determine if there is a consistent relationship between phosphate and DOC.

Phosphate has also been shown to be more soluble in sodic soil compared to non-sodic soils (Jalali and Merrikhpour, 2008). Soils with higher concentrations of Na^+ ions had higher leaching rates of Ca^{2+}, Mg^{2+}, K^+, and P and higher adsorption rates of Na^+

(Jalali and Merrikhpour, 2008). Reduction in phosphate adsorption may be attributed to reduced electrostatic potential which, in turn, reduces the adsorption ability of phosphate to soil (Barrow, 1985; Steele and Aitkenhead-Peterson, 2012b). When Na^+ replaces other cations on the CEC, it may make soil charge more neutral, causing a decreased attraction between phosphate and soil, ultimately causing increased mobilization of phosphate from soil.

1.5.2 Nitrate and ammonium

Nitrate is an extremely mobile ion and is readily transported through the soil column to groundwater sources or through runoff to stream water. Excess nitrate is in urban streams leads to serious environmental issues, (Burford and Bremner, 1975), including poor drinking water quality, eutrophication, and hypoxia (Groffman et al., 2005; Mitsch et al., 2001). Sources of excess nitrate are typically WWTP effluent, sewage and nitrate fertilizers (Groffman et al., 2005). Management strategies for nitrate currently include decreasing nitrate sources and increasing inorganic nitrogen sinks (Groffman et al., 2005; Mitsch et al., 2001). Nitrogen sinks include autotrophic uptake (bacteria and plankton), heterotrophic immobilization and denitrification (Groffman et al., 2005), which can occur in wetlands and riparian buffers (Harrison et al., 2011; Mitsch et al., 2001).

Merely reducing nitrogen loading is not sufficient to reducing nitrate pollution. Mitsch et al. (2001) suggests a 2 tiered approach: (1) reducing nitrogen loading and (2) understanding how nitrogen is transported and interacts throughout the waterbody. The latter is paramount in further reducing hypoxia and eutrophication. Rivers generally

stretch hundreds of kilometers and pass through multiple states (Mitsch et al., 2001). Efficient management of non-point sources of nitrogen between states is an extremely difficult process, which is why understanding nitrogen interactions is an important management strategy.

There are varied research observations related to the interaction of DOC and nitrate. Multiple studies found the denitrification rates were higher in urban streams due to the presence of higher quality organic matter (Groffman et al., 2005; Newcomer et al., 2012), the source of which is likely grass clippings and increased non-point sources of nitrate compared to undisturbed ecosystems (Newcomer et al., 2012). Increased concentrations of DOC, act as an oxygen source for microorganisms and serve as an electron donor during denitrification (Newcomer et al., 2012; Seitzinger, 1994) ultimately resulting in an increase in denitrification and decrease in nitrate (Burford and Bremner, 1975; Groffman et al., 2005).

There have been studies which have not seen the same increase in denitrification (Bernhardt and Likens, 2002; Groffman, 1999). Bernhardt and Likens (2002) attributed this to a filamentous mat of bacteria intercepting DOC before it made it to stream sediment. Without DOC reaching the anoxic zone, it would have no effect on denitrification, as that is where it typically occurs. Bernhardt and Likens (2002) also observed a decrease in nitrification potential and nitrate concentrations. They associated this trend with increasing C:N ratio which provided increased competition for heterotrophic bacteria and nitrifiers due to DOC addition. Because nitrifiers are not as strong of a competitor for nitrate, there was a reduction in nitrification (Bernhardt and

Likens, 2002). There was also a subsequent increase in demand for ammonium. Ultimately, this study observed decreased ammonium and nitrate in the environment. Another study which did not observe increased denitrification was (Groffman, 1999). Researchers added carbon to soil which resulted in a decrease in total nitrogen and increase in inorganic nitrogen; however, they were expecting to observe an increase in total C and N and decrease in inorganic nitrogen (due to increased denitrification). They hypothesized this observed trend was either due to (1) only an increase in microbial respiration rate with no population growth or (2) increased microbial activity and growth which was fed on by nematodes and protozoa. Reason 2 was supported by an observed increase in mineralization, as is typically seen by increased protozoa and nematode activity (Groffman, 1999). The wide variety of research findings is representative of the need for additional research on the relationship between DOC and the nitrogen cycle.

The relationship between sodium and the nitrogen cycle has not been widely studied. Duckworth (1991) observed increased ammonium mobilization from soil and a decrease in ammonium retention from atmospheric deposition. There were also decreased concentrations of nitrate, potentially due to the acidic nature of the forest soil (pH~ 4-7) (Duckworth, 1991). pH has an indirect influence on the nitrogen cycle by altering both ammonification and denitrification rates (Green et al., 2008a). Denitrifying microorganism activity is markedly reduced in acidic soil conditions thereby reducing mineralization and nitrification. Conversely, under more neutral or basic conditions, those rates would be increased potentially resulting in increased mobilization of ammonia and nitrification from the soil (Green et al., 2008a).

1.6 Drivers for increased DOC in Northern Hemisphere surface waters

Dissolved organic carbon concentrations in freshwaters in the northern hemisphere have been increasing at an unprecedented rate (Evans et al., 2006; Evans et al., 2005; Monteith et al., 2007), causing a potential disruption in the natural carbon cycle. Any variation in the carbon cycle is of great importance to researchers in order to determine any implications on the global carbon cycle and concentrations of atmospheric CO_2. There is natural variability in DOC concentrations influenced by changes in precipitation, hydrological flowpaths, season, adsorption to or desorption from soil minerals, and microbial processes; however, recent studies have found that these natural variabilities only account for a small percentage of the regional increases in DOC and there are other drivers influencing this trend (Aitkenhead-Peterson et al., 2009; Evans et al., 2006; Evans et al., 2005; Monteith et al., 2007; Raike et al., 2012). Some of these hypotheses include: climate change, decreased acid deposition, increased urbanization or land use change, steadily increasing atmospheric CO_2. More recently increased sodium to watershed soils has been postulated as a driver of increased riverine DOC (Aitkenhead-Peterson and Steele, 2016; Aitkenhead-Peterson et al., 2009; Green et al., 2008b), particularly in human-dominated watersheds.

Average DOC concentrations in rivers has been determined to be 5.8 mg L^{-1}, but can vary dramatically (<1-50 mg L^{-1}) (Thurman, 1985). Despite the natural variation in DOC concentrations, multiple studies have observed an upward trend in DOC. A study in the UK found DOC concentrations at 22 river sites had increased an average of 91% over a 15 year time period (Evans et al., 2005). Total organic carbon (TOC) flux has

increased by 38% in Finnish rivers (Lepistö et al., 2008). North America has seen similar trends; DOC concentrations in 7 out of 48 lakes in the Adirondack region have increased significantly since monitoring began (Driscoll et al., 2003). Two separate studies found increasing DOC concentrations throughout lakes in the Ontario and Quebec regions of Canada (Bouchard, 1997; Houle et al., 2004). An EPA report found DOC concentrations to increase 10% (0.05 mg L^{-1} yr^{-1}) in 4 out of 5 studied regions; however, they hypothesized that DOC increases may be a sign of environmental recovery in response to the Clean Air Act, which reduced the release of sulfur aerosols into the atmosphere (Stoddard et al., 2003).

Other researchers have postulated reasons why DOC is increasing, specifically in watersheds that have increased urban and suburbanization. The drivers of increased aquatic DOC include deicing salts (NaCl) and use of sodic groundwater ($NaHCO_3$) for irrigation purposes. While oceanic deposition (sea salt) is the same sodium form as deicing salts (NaCl), it has been associated with declines in DOC and DON as well as pH.

1.6.1 Interaction between sodium cations and DOC and DON

Many studies have recognized a relationship between Na cations and increased DOC desorption (Cooper et al., 2014; Green et al., 2008a; Green et al., 2008b; Kim and Koretsky, 2012; Steele and Aitkenhead-Peterson, 2012a). Excess Na concentrations result in an ion exchange with Ca^{2+}, Mg^{2+}, and K^{+} on the CEC (Löfgren, 2001; Norrström and Bergstedt, 2001; Shanley, 1994). Typically, Ca^{2+} will be displaced first, followed by K+ and Mg2+ possibly due to slower supply and restoration rates

(Norrström and Bergstedt, 2001). This can cause soil nutrient deficiencies as well as sodic soils resulting from high Ca and Mg concentrations. Sodic irrigation water has been linked to increased desorption of DOC from soil (Aitkenhead-Peterson and Cioce, 2013). Na^+ can also displace H^+ ions, increasing soil pH (Stevenson, 1994). As DOC is highly soluble in high pH, it enhances DOC and DON desorption.

Excess Na can also cause increased colloid dispersion in soil due to high exchangeable sodium percentage (ESP) and soil solution salt concentration (Green et al., 2008b; Norrström and Bergstedt, 2001). High ESP and salt soil solution concentrations are known to mobilize heavy metals into surface waters (Green et al., 2008b; Norrström and Bergstedt, 2001) and have also been shown to increase mobilization of DOC and DON (Green et al., 2008b). Therefore, the impact of sodium and it's interaction with cations on DOC concentration is threefold: (1) through exchange with Ca^{2+} and Mg^{2+} on CEC causing sodic soils, (2) displacement of H^+ ions causing increases in soil solution pH and solubilization of DOC, and (3) enhanced colloid dispersion and mobilization of DOC.

1.6.2 Deicing salts

Deicing salt use has continually increased since the 1940's in the United States, Canada and Europe. Currently, the United States uses over 15,000,000 tons of deicing agents annually compared to 164,000 tons in the 1940's (Kim and Koretsky, 2012). NaCl is the most commonly used deicing salt, with $CaCl_2$ being more expensive and typically used on a smaller scale by homeowners (Green et al., 2008b; Kim and Koretsky, 2012). Deicing salts are often mixed with grit or sand and, less often, mixed

with compounds such as sodium hexacyanoferrate II for anti-caking purposes (Green et al., 2008a). There are many negative environmental impacts associated with deicing salts including decreased aquatic biodiversity, declining amphibian health, increasing sodium content of potable water supplies, increasing colloid mobility, and altering cation composition of cation exchange sites resulting in changes of the structure and natural biogeochemical cycles in soil (Green et al., 2008a; Kim and Koretsky, 2012).

Long term use of sodium on DOC and DON in soils was investigated by Green et al. (2008b). Organic matter content and concentrations of DOC and DON in solution changed depending on the length of exposure to deicing salts. Control soils and soils exposed to deicing salts for shorter periods of time (<10 years) were shown to have a greater response to salt addition and increased mobilization of DOC and DON from soil. Soils that have been exposed to salts for longer periods of time released less DOC and DON, assumedly because a larger proportion of soluble organic matter had already been mobilized from the soil. Green et al. (2008b) attributed this temporal effect to sodium's influence on soil aggregate dispersal; sodium competes with Ca^{2+} and Mg^{2+} on exchange sites which decreases soil stability by removing the bonds between polyvalent cations and organic matter (Cooper et al., 2014; Green et al., 2008b). Addition of sodium also changes ion dominance of cation exchange capacity (CEC); H^{+} ions on cation exchange sites are replaced by Na^{+}, thus increasing pH and organic matter solubility. Increasing aggregate instability and pH allows for enhanced desorption of DOC and DON from the soil, until the soluble organic matter threshold is reached. At that point, less DON and DOC is released because most of the soluble forms have already been mobilized from

the soil; this is the basis for Green et al.'s (2008b) *"when it's gone, it's gone hypothesis"* which states that (1) increasing organic matter mobilization over the years has removed soluble forms from the soil and (2) enhanced sodium concentrations prevent vegetation growth and negates potential additional input of soluble organic matter in the soil.

1.6.3 Sodic irrigation water

Fewer studies have been conducted on DOC concentrations in the southern portion of the United States, with most studies focused on states in the northern hemisphere affected by deicing salts and sulfur deposition. However, Texas has similarly high concentrations of DOC in urban streams, yet uses very little, if any, road deicing salts (Steele and Aitkenhead-Peterson, 2012b). Multiple studies conducted on DOC concentrations in Texas have found a correlation between increasing urbanization and DOC concentrations (Aitkenhead-Peterson and Steele, 2016; Cioce and Aitkenhead-Peterson, 2015; Steele and Aitkenhead-Peterson, 2011; Steele and Aitkenhead-Peterson, 2012a; Steele and Aitkenhead-Peterson, 2012b). These studies have hypothesized that increasing urbanization is associated with excess loading of sodium and chloride into the environment, resulting in the leaching of allochthonous DOC into the aquatic environment. Steele and Aitkenhead-Peterson (2012b) found that population density was a predictor of sodium loading and could account for 13% of total sodium.

Potential sources of significant sodium loading in urban areas has been linked to waste water treatment plant (WWTP) effluent and urban open spaces (Aitkenhead-Peterson et al., 2009). Multiple studies have linked WWTPs to high concentrations of DOC in waterways, but in watersheds lacking WWTPs Aitkenhead-Peterson et al.

(2009) found that 68% of the variability could be attributed to urban open areas such as golf courses, neighborhoods and sports fields. Steele and Aitkenhead-Peterson (2011) found this could be explained by the sodicity of irrigation water used; for example, sodium adsorption ratio (SAR) and total soil N accounted for 71% of DOC variability. Irrigation water is considered a low priority use of water and its quality is generally low and in some instances recycled waste water, industrial effluent or brackish groundwater are used (Steele and Aitkenhead-Peterson, 2011). Sodium ions (Na^+) did not account for an increase in DOC alone, it was the ratio of Na^+ to Ca^{2+} and Mg^{2+} as either $\%Na^+$ or sodium adsorption ratio (SAR) (Steele and Aitkenhead-Peterson, 2012a; Steele and Aitkenhead-Peterson, 2012b). Steele and Aitkenhead-Peterson (2012a) concluded this may be due to SAR measuring the probability of cations, such as sodium, interacting with functional groups on organic compounds. As such, when SAR increases, sodium is more likely to interact with the functional groups, causing the increased solubility and subsequent desorption of DOC from the soil, allowing it to run off and increase surface water concentrations of DOC.

1.6.4 Sea salt and acid deposition

Multiple studies have attributed the rise in DOC in glaciated, acid-sensitive environments in the northern hemisphere to decreased acid deposition from SO_4^{2-} (Evans et al., 2006; Evans et al., 2005; Monteith et al., 2007). Europe and areas of eastern North America have seen declines in sulfur deposition from acid rain after initiatives to decrease air pollution, such as the Clean Air Acts, in Europe and the United States. Simultaneously, Europe saw less oceanic sea salt deposition due to increased storm

activity (Evans et al., 2006; Evans et al., 2005; Monteith et al., 2007). Two mechanisms or hypotheses have been put forth to account for increased DOC observed in northern hemisphere surface waters:

1) Acid addition to soils reduces the release of organic matter, which would account for lower DOC concentrations in the environment (Evans et al., 2005). This fundamental concept is based on F. J. Stevenson's work (1994) which showed that fulvic acids are soluble in low pH solution but humic acids are not. As pH increases in solution, humic acids are solubilized.
2) Declining acid deposition rates would increase DOC solubility and its presence in surface waters (Evans et al., 2006; Evans et al., 2005; Monteith et al., 2007). The concept is based on ionic strength reduction in soils (e.g. SO_4^{2-}, Ca^{2+}, Mg^{2+}) which would increase desorption of DOC (Evans et al., 2006; Evans et al., 2005; Monteith et al., 2007).

Conversely, work by Aitkenhead-Peterson and Steele (2016) showed that increasing ionic strength increased DOC concentrations in the tributaries of the upper Trinity River in Texas, USA. There is no definitive explanation for these seemingly contradictory results. It is likely that changes in DOC concentrations are not globally congruous, due to the variety of climates, soil types, non-point sources of pollution and ecological functions.

Environments responding positively to the acid deposition hypothesis are typically glaciated, acid sensitive, forested areas that overlay granitic bedrock and have little buffering capacity. However, DOC has been increasing in areas that do not meet

these criteria. Much of the research conducted on sulfur deposition in the North America has focused on northern, glaciated regions, specifically New England and Canada. Most of these sites were located in forested areas dominated by conifer species which have been shown to scavenge acidic atmospheric aerosols (Miller et al., 1993).

Monteith et al. (2007) postulated that declines in oceanic deposition could also be responsible for increased aquatic DOC. Few studies have examined this (Compton and Church, 2011). The mechanism is similar to acidic deposition in that oceanic deposition acidifies watershed soils, rendering DOC less soluble. With declines in oceanic deposition, pH of watershed soils would increase, subsequently solubilizing DOC. Generally, watersheds within 5-7 km from the ocean will be most affected by this phenomenon (Compton and Church, 2011). Low concentrations of NaCl (0.01 and 0.1 mM) mobilized more DOC than high concentrations of NaCl (1 mM) which suggests that declines in oceanic deposition, or more precisely reduction in their concentrations, would increase DOC mobility. This could be due to the study soils being primarily Andisols; salt addition to Andisols causes a decline in solution pH which may counteract DOC mobilization because DOC is more soluble at higher pH. Other studies found that the replacement of H^+ with Na^+ on soil receptor sites also cause an increase in pH (Green et al., 2008b).

1.7 Study objectives

The objectives of this study were:

1) To determine the temporal effects of exposure to sodium from different sources on water extractable, NO_3-N, NH_4-N, PO_4-P, DON and DOC in urban soils.

H_o: There will be no effect of sodium source or length of exposure on water extractable DOC, DON and nutrients.

H_1: Sodium exposed soils will have increasing concentrations of DOC and DON due to desorption from soil. Soils exposed to sodium longer than 20 years will have decreased concentrations of water extractable DOC and DON compared to similar soils with less than 20 years of exposure.

H_2: Sodium source will have an effect on the desorption rate of DOC and DON

2) To determine the effects of 2 and 4 hour shake times on measured concentrations of water extractable, NO_3-N, NH_4-N, PO_4-P, DON and DOC.

Ho: The measured concentrations of NO_3-N, NH_4-N, PO_4-P, DON and DOC will not be significantly different between shake times.

H_1: The measured concentrations of NO_3-N, NH_4-N, PO_4-P, DON and DOC will be significantly different between shake times.

CHAPTER II

THE IMPACT OF URBAN SODIUM INPUTS ON WATER EXTRACTABLE DOC AND DON

2.1 Methods

2.1.1 Site descriptions

Samples were collected from Frederick, MD (n=35), Bryan-College Station, TX (BCS) (n=34), and Galveston, TX (n=35). Among the 3 cities soil (Table 2) and climate characteristics (Table 3) varied. Samples from Frederick were collected to determine the effect of sodium from deicing salts (NaCl) (Table 4). Records for salt use in Frederick, MD began in 2004 (Table 5) and varies from year to year based on snowfall. There are no records on when salting began, but it has been used since at least the 1970's when it was a mixture of 20% salt and 80% ash. In the early 2000's the mixture changed to salt and stone chips. As of 2014, the mixture includes 100% NaCl and no anti-skid components (Ramsburg, 2015).

Galveston samples were collected to provide insight into the effect of oceanic deposition (NaCl) (Table 4). Oceanic deposition of sodium has produced a very slight 1% increase since 1984 according to the National Atmospheric Deposition Program (NADP). NADP records of atmospheric sodium deposits showed deposition to range from 2.5 to 6.0 kg/ha annually in Colorado County, near the Gulf Coast. Bryan-College Station samples were collected to examine the impact of salt ($NaHCO_3$) in lawn

irrigation water on release of DOC, DON and nutrients from soil (Table 4). The cities of Bryan and College Station use a significant volume of municipal water for landscape irrigation (White et al., 2004). This water has high concentrations of sodium relative to municipal water in the other cities examined (Table 6).

Table 2: Soil groups and soil attributes for the cities sampled in this study.
OM = organic matter, Ksat = saturated hydraulic conductivity, BD = soil bulk density, CEC = cation exchange capacity. *Source: (Natural Resource Conservation Service and University of California Davis)*

City	Soil Group	Sample Size	Clay	Sand	Silt	OM	Ksat	BD	CEC
		%	%	%	%		mm/hr	g/cm^3	cmol charge /kg soil
Frederick	Alfisol	54.3	18±5	30±8	52±4	2±1	48±26	1.3±0.1	13±4
	Inceptisol	14.3	17±4	38±7	45±9	2±1	60±37	1.4±0.1	15±4
	Ultisol	31.4	19±2	40±5	41±6	2±0	31±7	1.4±0.1	13±2
Galveston	Entisol	74.3	5±0	94±0	1±0	1±0	331±0	1.6±0	3±0
	Beach	25.7	2±0	98±0	1±0	0±0	508±0	1.5±0	3±0
BCS	Alfisol	97.1	11±2	68±7	21±6	1+0	44±53	1.5±0.1	5±1
	Vertisol	2.9	50±0	22±0	28±0	2±0	0.8+0	1.8±0	50±0

Table 3: Climate characteristics of 4 cities examined for this study.

Site	Avg. Annual Precip. (mm)	Avg. Annual Snowfall (mm)	Avg. Max Temp. (°C)	Avg. Min. Temp. (°C)
Bryan-College Station, TX	1017.5	0	26.2	15.0
Galveston, TX	1093.5	0	24.7	17.2
Frederick, MD	1110	406.4	17.2	5.7

Source: (Arguez et al., 2010)

Table 4: Predominant sodium source and demographics of the three cities examined.

City	Sodium Source	Settled Year	Area km^2	Population #	Density # km^{-2}
‡Bryan/College Station, TX	Irrigation water	1866	115	228,660	926
‡Frederick, MD	Deicing salts	1745	60	65,239	1145
‡Galveston, TX	Oceanic deposition	1816	539	47,243	479

Climate (Koppen-Geiger) ‡Cfa.

Table 5: Amount of road salt used in Frederick, MD since 2004.

Fiscal Year	Amount Used (Tons)
FY 04	12,622
FY05	14,098
FY06	10,231
FY07	19,181
FY08	17,663
FY09	11,282
FY10	13,853
FY11	12,393
FY12	5,419
FY13	14,191
FY14	31,712
FY15	29,175
Total:	191,820
Average:	15,985

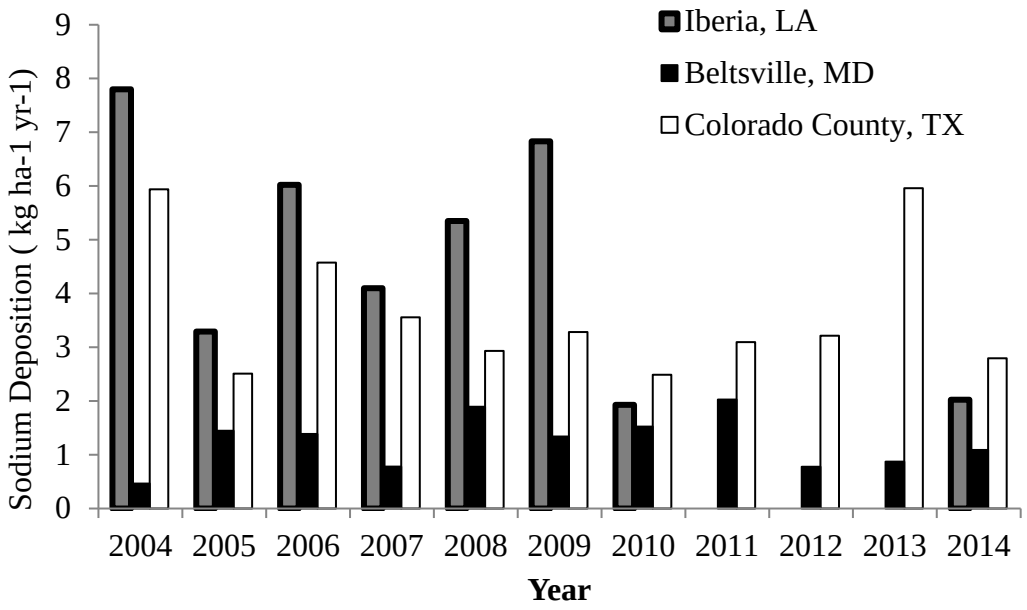

Figure 1: Atmospheric sodium deposits for NADP sites. Monitoring stations are similar distance from the coast as the cities studied were selected. Frederick, MD is 110 miles from the coast and Beltsville, MD (80 miles from the coast was selected as its NADP station). Galveston is 0 miles from the coast and New Iberia, LA (37 miles from the coast) was selected as its NADP station) Bryan/ College Station is 130 miles from the coast and the Attwater Prairie Chicken National Wildlife Refuge in Colorado County (75 miles from coast) was selected as its NADP Station. *Source of Data: (NADP Program Office et al., 2015).*

Table 6: Sources of municipal water and associated sodium concentrations for each of the cities examined.

City	Year	Source	Waterbody	Na^+ mg L^{-1}
Galveston, TX	2015	Surface	Brazos River	47- 63
Bryan, TX	2011	Ground	Simsboro Aquifer	230
College Station, TX	2011	Ground	Carrizo Wilcox/Sparta Aquifers	193
Frederick, MD	2014	Surface	Linganore Ck, Monocacy River	nd

No data=nd. *Source: (City of Bryan, 2014; City of College Station, 2014; City of Frederick, 2015).*

2.1.2 Experimental design

Soils were collected within three feet of the road from two landscape positions: 1) single-family home lawns and 2) roadsides from three cities in the USA exposed to the different sodium sources. Roadside refers to the grass strip in between the road and the sidewalk. Samples from roadsides were hypothesized to be important because there was a greater expectation of an affect from deicing salts closer to the road when compared to lawns. Exposure to sodium from oceanic deposition and sodic irrigation was not expected to differ when comparing landscape position. The turf placed during home construction was also assumed to have not been previously exposed to sodium. Therefore, soils collected from homes at age range 0-5 were treated as the control samples.

In Frederick, sites were chosen by cross-referencing roads under county jurisdiction for deicing salt application. Homes were found by using websites posting homes for sale (e.g. www.Zillow.com). In Bryan-College Station, sites were chosen by determining if they had irrigation systems. This information was obtained by looking at websites posting homes for sale (e.g. www.realtor.com). In the case of BCS, roadside sites adjacent to homes with irrigation were assumed to also have irrigation systems. There was no information about the history of each home's irrigation system; it was assumed to be present at the site since initial construction. However, the older single family homes were unlikely to have had in-ground irrigation systems when constructed but had in-ground irrigation systems when sampled. Galveston sites were chosen by getting a sample set representative of the length of the island.

To assess time of exposure, an assumption was made that grass was provided as sod when the home or road was constructed or, if seeded, top soil was obtained from a location unlikely to have experienced Na^+ exposure. An equal distribution of samples was taken from age ranges of a) 0-5 years, b) 6-10 years, c) 11-20 years, d) 21-30 years and e) greater than 30 years.

2.1.3 Sample collection and processing

Three soil samples of 15 cm depth were collected at each site using a 2 cm diameter soil probe and composited. Soils were air dried immediately after collection and sieved using a 2 mm sieve when dry.

2.1.4 Water extraction of soils

DOC, total dissolved nitrogen (TDN), NO_3-N, PO_4-P, NH_4-N), pH and EC, extracts underwent a 1:10 soil:ultrapure extraction and were shaken for 2 hr. For base cations and metals, soils were also extracted at a 1:10 ratio but shaking time was increased to 4 hr.

For the 2 hour shake extracts, 50 mL high density polyethelene (HDPE) centrifuge tubes were filled with 3 g of soil and 30 mLultrapure water. After 2 hours of shaking, samples were centrifuged at 19,000 g-force for 15 minutes at room temperature. For the 4 hour shake extracts, 250 mL HPDE bottles were filled with 25 g of soil and 250 mL of ultrapure water. After 4 hours of shaking, samples were centrifuged at 19,000 g-force for 5 minutes at room temperature. The extracts were filtered through Whatman GF/F filters (0.7 µm nominal pore size) to remove any remaining floating organic particles prior to analysis.

2.1.5 Chemical analysis

A high-temperature platinum-catalyzed combustion with a Shimadzu TOC-V_{CSH} and Shimadzu total measuring unit (Shimadzu Corp. Houston, TX, USA) was used to measure DOC and TDN. USEPA method 415.1 was used to measure DOC by acidifying the sample and sparging it for 4 min using C-free air.

The nutrients NO_3-N, NH_4-N, and PO_4-P were measured using Westco Scientific Smartchem Discrete Analyzer (Westco Scientific Instruments Inc. Brookfield, CT, USA). USEPA method 353.3 was used to measure NO_3-N, utilizing the Cd-Cu reduction method and USEPA method 350.1 was used to measure NH_4-N utilizing the phenate hypochlorite method with a sodium nitroprusside enhancement. PO_4-P was quantified as orthophosphate-P using the ammonium molybdate method (USEPA 365.1). DON is estimated as the difference between TDN and the sum of NO_3-N and NH_4-N.

For the analysis of water extractable soil base cations (Ca^{2+}, $Mg^{2+,}$ K^+ and Na^+) and metals (Fe, Mn, Cu, B, Zn, Mg, and S), soil-water extracts (1:10) that had been shaken for 4 hr were analyzed using inductively coupled plasma mass spectrometry (ICP) (Spectro Genesis: Spectro, Germany) using the method described in (Franson, 1989).

2.1.6 Statistical analysis

Average concentrations, standard deviations, and standard errors were calculated for each sodium source and age group for every water extracted nutrient. All data was also tested for normality. Univariate analysis of variance was performed in turn with each extract type (2 hr vs 4 hr shake times) as the dependent variable and a) sodium

source, and b) time of exposure to sodium source as independent factors. Age group was used as an independent factor to assess significant effects on water extractable nutrients. Landscape position was not included in the univariate analysis because it was determined using a two-way ANOVA that landscape position had no significant effect on DON, DOC, PO_4-P, NO_3-N, and NH_4-N thus the data was pooled thereafter resulting in a larger sample size for each sodium source and age grouping. Regression analysis was used to examine relationships between dependent and independent variables. A backwards regression analysis was conducted to determine the best independent variables for predicting DOC and DON. The variables from the backwards regression model were then used in The Unscrambler v 9.8 (Camo Software Inc., Woodbridge, NJ, USA) where a multiple regression analysis with a full cross validation was run.

2.2 Results

2.2.1 pH, DOC and DON

There was no significant difference when comparing BCS and Galveston pH in the 0-5 year exposure group (Figure 2). By age range 6-10 years, pH was significantly different among the cities, with pH beginning to show a decline in Galveston and increase in BCS. In the 0-5 year exposure group for Frederick, pH was different from Galveston and BCS, and did not change overtime. By age 6-10, Galveston and Frederick pH were no longer significantly different.

DOC in both Frederick and Galveston was not significantly different between cities or over time (Figure 2). DOC in BCS from age 0-5 and 6-10 was not different

from either Galveston or Frederick; however, by age 11-20, concentrations differed significantly from both Frederick and Galveston (Figure 2). This implies that irrigation water has a more significant impact on DOC desorption than deicing salts and oceanic deposition. While Frederick had no significant changes in DOC, it followed a trend similar to BCS in that DOC peaked at 21-30 years with concentrations declining after 30 years. This is in line with Green et al. (2008b) "*When it's gone, it's gone*" hypothesis which stated increasing organic matter mobilization over the years has removed soluble forms from the soil, therefore, causing an eventual decline in DOC mobilization. Contrary to BCS and Frederick, DOC in Galveston appeared to have a more linear relationship.

DON followed the same pattern as DOC. It did not change over time in Frederick or Galveston. Concentrations in BCS were not significantly different from Frederick or Galveston until age 21-30. There was also a linear increase in DON in Galveston. DOC, DON and pH were also positively correlated with each other (Figure 3). pH explained 85% and 64% of the variability in DOC and DON, respectively (Figure 3).

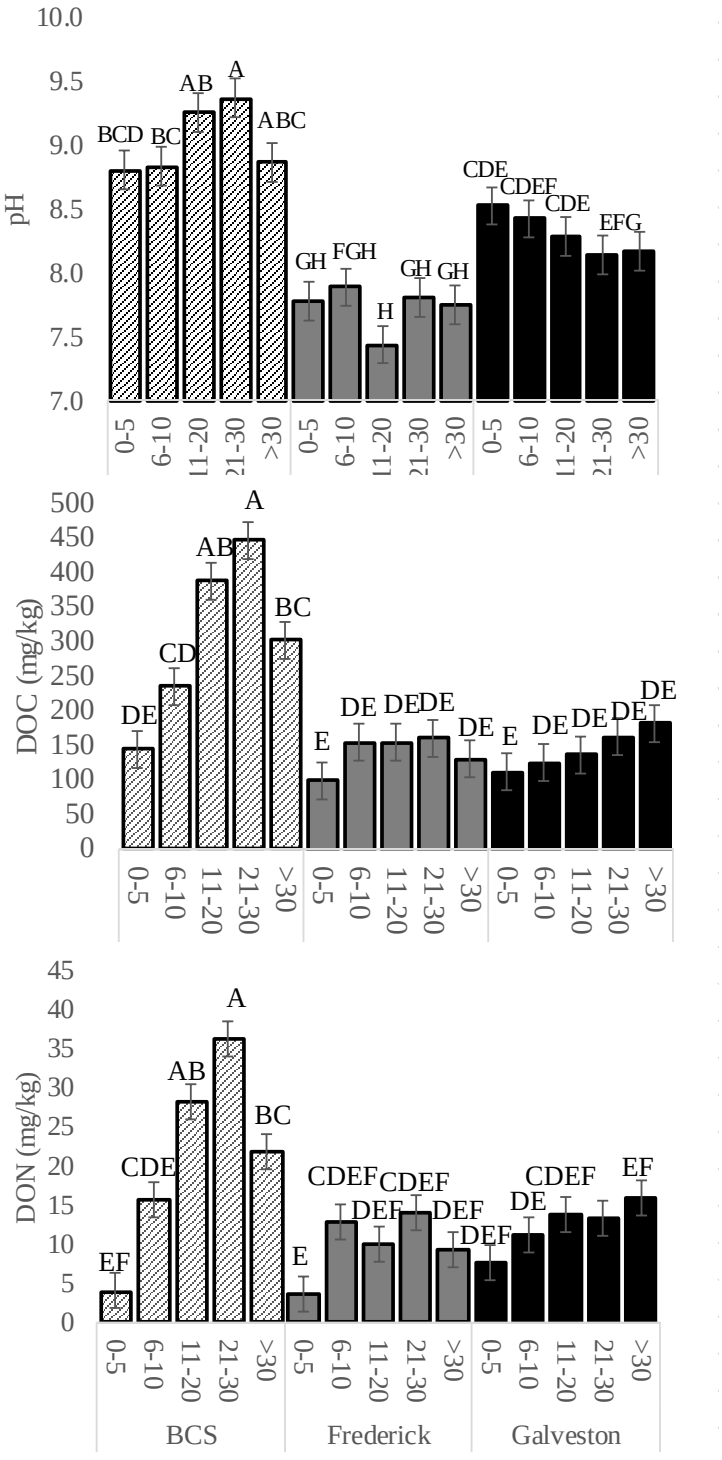

Figure 2: Mean pH, DOC and DON for Frederick, Galveston and BCS. Different uppercase letters signify significant difference when comparing columns. $\alpha < 0.05$.

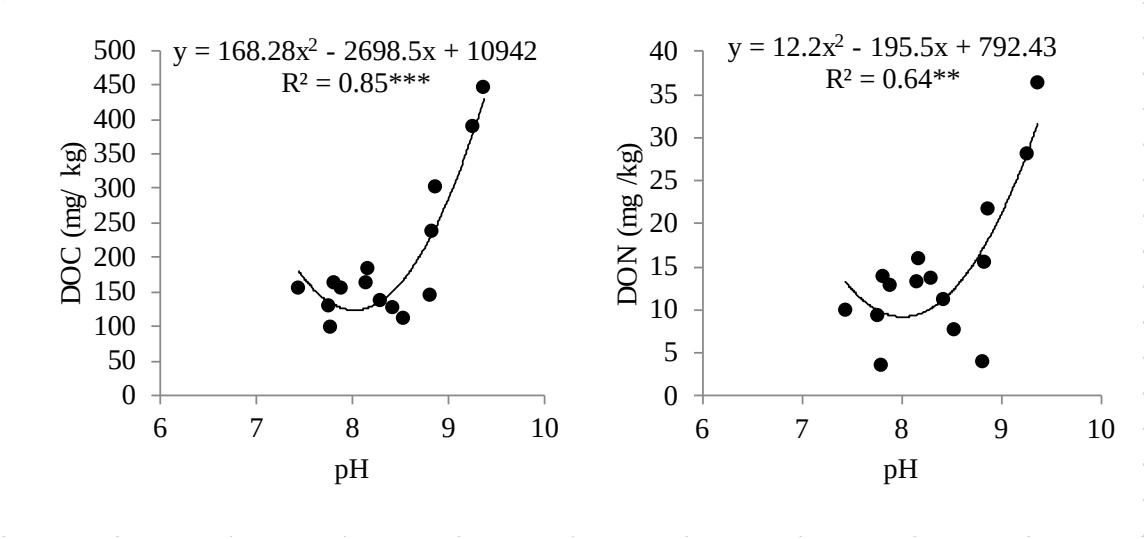

Figure 3: Relationship between DOC, DON and pH. Each point represents the mean of each exposure time for each city.*** represents p-value <.0001 and ** represents p-value of <.01.

A backward, stepwise, multiple linear regression model was created for the estimation of DOC and DON (Figure 4). The most significant model for DOC was:

(1)

$$DOC = -80.32 + (18.73 * Na\ Source) + (10.42 * pH) + (0.11 * EC) + (2.4 * NH_4 - N) + (-4.1 * PO_4 - P) + (8.9 * DON) + (0.68 * Mg) + (-78.33 * B) + (.05 * Fe) + (29.5 * Zn) + (-33.77 * Mn).$$

This model explained 93% of the variability of DOC. When observed and estimated (full cross-validation) DOC values were graphed with the regression line intercepting 0, the R^2 reduced to 0.89 ($p < .0001$) (Table 7). According to this model, DOC prediction was most influenced by DON, pH and sodium source (Table 2.7)

For DON the most significant model was observed to be

(2)

$$DON = -1.96 + (0.05 * Age\ Range) + (2.15 * Na\ Source) + (-0.04 * NO_3 - N) + (0.73 * PO_4 - P) + (-0.04 * Mg) + (0.01 * Na) + (-1.78 * Zn) + (4.03 * Mn) + (0.09 * DOC)$$.

The model explained 89% of the variability in DON ($p < .0001$). When observed and estimated DON values were graphed with the regression line intercepting 0 the R^2 was reduced to 0.87 ($p < .0001$). (Table 7). According to this model, DON prediction was most influenced by DON, sodium source and phosphorus (Table 2.8).

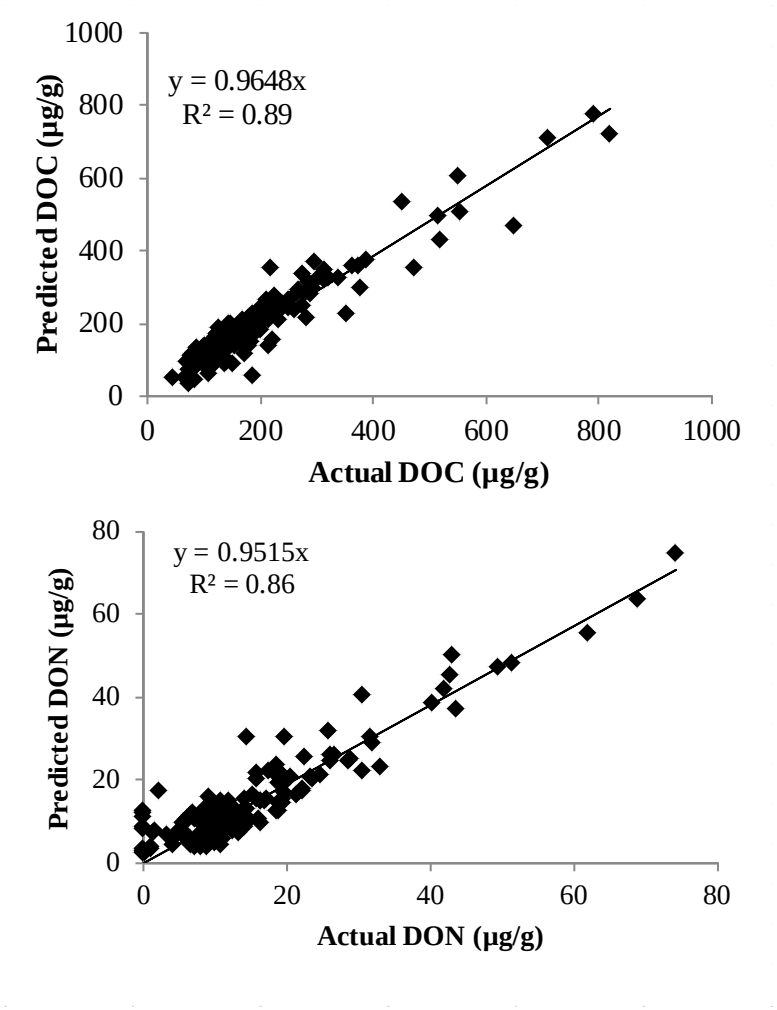

Figure 4: Observed and predicted DOC and DON concentrations for each sample. Cross validation was performed using The Unscrambler v 9.8 software.

Table 7: Values for DOC and DON cross validation models.

	Slope	RMSE	R^2 model	Adjusted R^2 model	R^2 Cross Validation	P-Value
DOC	0.9648	29.89	0.97	0.97	0.89	<.0001
DON	0.9515	3.03	0.97	0.97	0.86	<.0001

Table 8: Standardized coefficients to determine which independent variables most influenced DOC desorption.

DOC	
DON	138
pH	87.5
Sodium Source	37.5
Zn	35.4
Mg	30.6
EC	12.7
Fe	11
NH_4	6.7
PO_4	-0.9
Mn	-16.9
B	-31.3

Table 9: Standardized coefficients to determine which independent variables most influenced DON desorption.

DON	
DOC	18.6
Sodium Source	4.3
PO_4	2.3
Mn	2.0
Na	1.5
Age Range	0.2
NO_3	-0.5
Mg	-1.8
Zn	-2.1

2.2.2 Sodium

Water extractable sodium concentrations were higher in BCS when compared to Frederick and Galveston (Figure 5). In fact, sodium appeared to be naturally higher in

BCS as evidenced by the high concentrations of sodium at age range 0-5. Although, if sod was grown in BCS it is likely did it did have previous sodium exposure. Na also varied significantly over time in only BCS (Figure 5). In BCS, sodium concentrations did not significantly differ from initial values until soil reached 21-30 years of age. This mirrors the pattern observed for pH, DOC and DON (Figure 2). While there were no significant differences in sodium for Galveston or Frederick, the same patterns were also seen in DOC, DON and pH. For Frederick, concentrations of sodium peaked at 21-30 years and began again to decline after 30 years of sodium exposure. In Galveston, sodium continually decreased over time, while DOC increased.

ESP was also much higher in BCS compared to the other cities (Figure 5). Initial ESP did not significantly differ over time in Frederick or BCS until exposure time to sodium was over 30 years (Figure 5). In Galveston, there was no significant difference in ESP but it did appear to decrease linearly over time.

As with sodium and ESP, SAR values were much higher in BCS (Figure 5). SAR also followed a pattern similar to DOC, DON and Na in BCS (Figure 5). For BCS and Frederick, SAR value did not vary significantly from the initial value until an exposure age of 21-30. For Galveston, there were no significant differences in the soil sodium metrics examined but values over time were lower than the initial value.

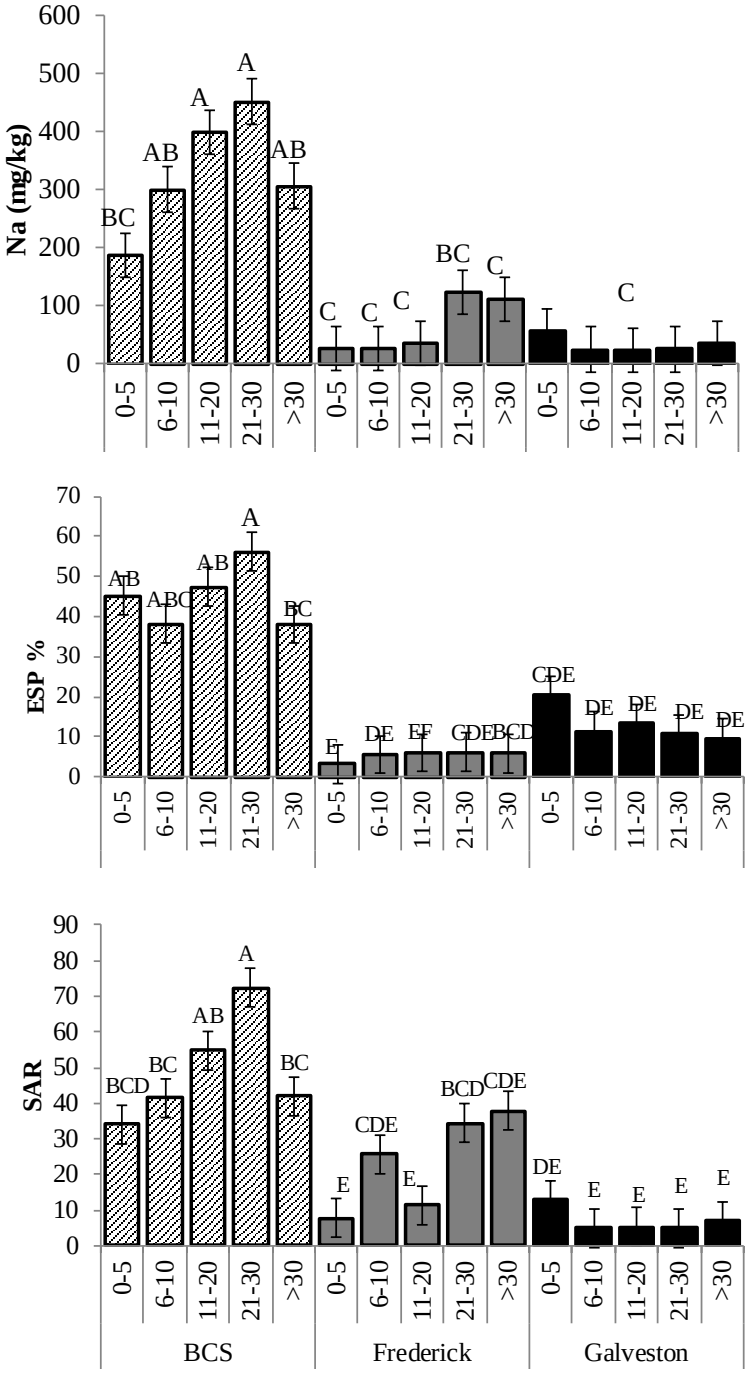

Figure 5: Mean Na^+, SAR and ESP for Frederick, Galveston and BCS. Columns with the same letter are not considered significantly different. For values which are significantly different, P<.05.

2.2.3 Nutrients

No significant differences in water extractable NO_3-N was observed in Galveston or BCS. In Frederick, water extractable NO_3-N concentrations were similar to both other cities but had noteworthy peaks in concentration at 11-20 yr and >30 yr which were significantly different from nitrate concentrations in soils in the 0-5 year range (Figure 6).

In Galveston there was no significant change in PO_4-P concentrations and no discernible pattern observed. In BCS and Frederick, PO_4-P became significantly different from initial concentrations at 21-30 yr (Figure 6). In Frederick, concentrations began to decline after 30 yr, while in BCS concentrations continued to increase.

There were no consistently discernible patterns in the cities for NH_4-N (Figure 6). In Frederick, the only significantly different value from the initial concentration was observed at 11-20 yr. After this time period, concentrations dropped to concentration observed for 0-5 yr. In BCS, concentrations of NH_4-N were significantly different from initial concentrations (0-5 yr) at ages 11-20 and 21-30. In Galveston, there were no significant differences and no apparent patterns in water extractable NH_4-N concentrations.

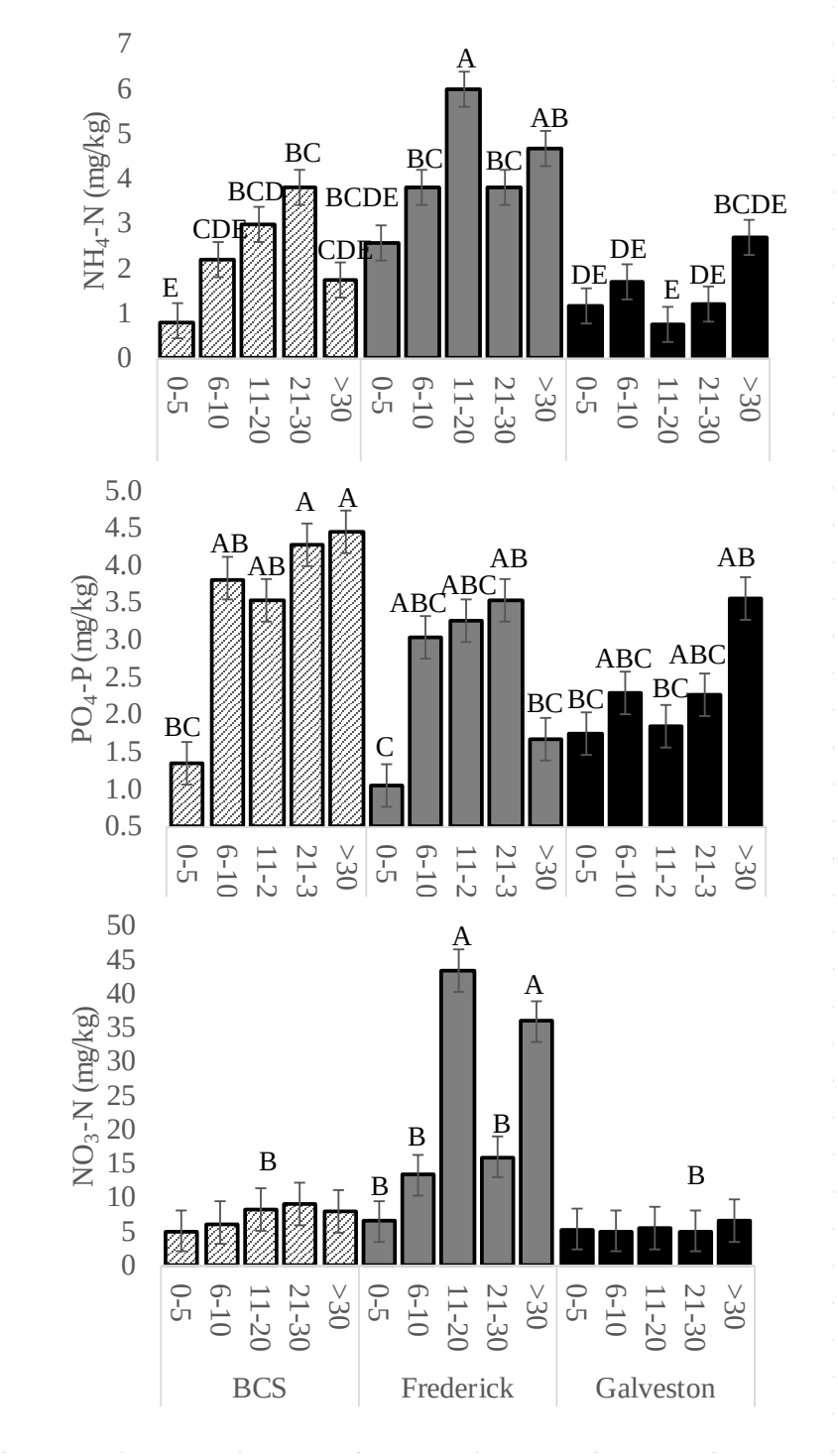

Figure 6: Mean NO_3-N, NH_4-N, and PO_4-P for Frederick, Galveston and BCS. Columns with the same letter are not considered significantly different. For values which are significantly different, P<.05.

2.2.4 Metals

Mg, B, Fe, Zn, and Mn were important variables for the prediction of DOC (Equation 1; Figure 7; Figure 8), and Mg, Zn, and Mn were important variables for the prediction of DON (Equation 2; Figure 7; Figure 8). While many additional cations were measured, they are not included in further analysis due to their insignificance in the DOC and DON models. All metals (Zn, Fe, B, Mn, Mg) were higher in BCS (Figure 7; Figure 8). There was no significant change in metals over time in Frederick or Galveston. All metal concentrations in BCS, with the exception of Zn, changed significantly from age 0-5 yr when compared to 6-10 yr. In fact, B, Fe, Mg, and Mn followed the exact patterns over time and increased significantly at 6-10 years, peaked at 21-30 years and declined at over 30 years. Zn was not significantly different from initial concentrations at 0-5 yr until soil age reached 21-30 years. While Zn differed from other metals in terms of when it significantly differed from initial (0-5 yr) concentrations, all metal concentrations peaked at 21-30 years. This is similar to what is observed with DOC and DON (Figure 2).

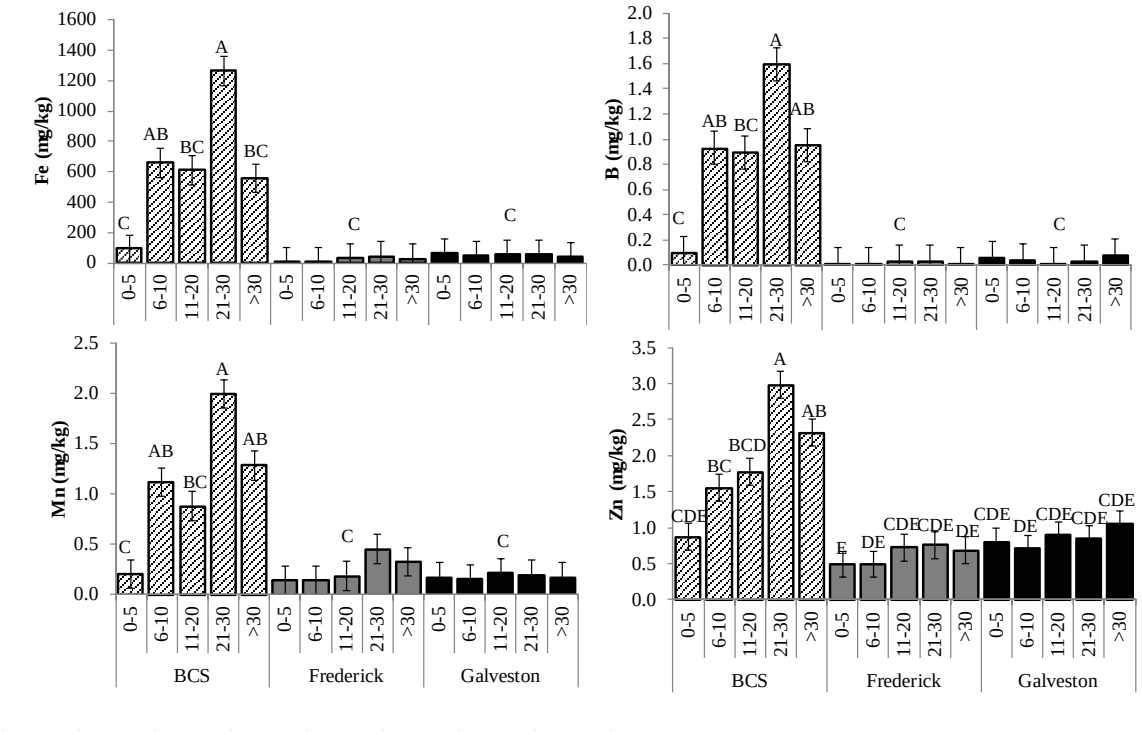

Figure 7: Mean Zn, Fe, B, and Mn for Frederick, Galveston and BCS. Columns with the same letter are not considered significantly different. For values which are significantly different, P<.05.

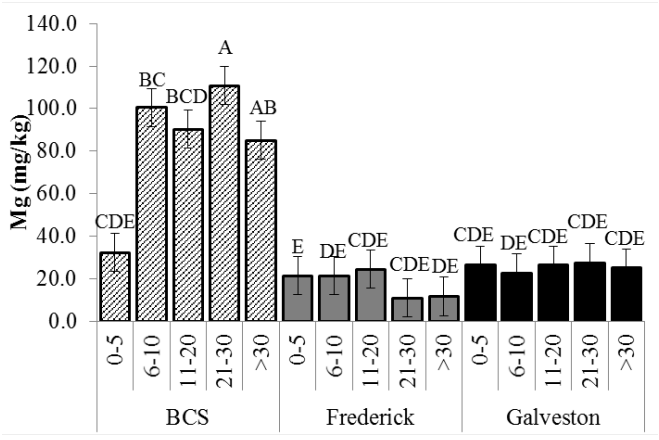

Figure 8: Mean Mg for Frederick, Galveston and BCS. Columns with the same letter are not considered significantly different. Values which are significantly different, P<.05.

2.3 Discussion

This study aimed to determine the temporal effect of sodium exposure on DOC and DON desorption in soils. This study investigated the effect of sodium source and time of exposure to sodium in an urban landscape as an alternative means of examining why DOC concentrations and exports might be increasing in surface waters. Three cities, each with a different sodium source from deicing salts, oceanic deposition or sodic irrigation were examined. In order to understand the mechanisms of DOC and DON desorption a model was also created to look at the independent variables that best predicted DOC and DON. A secondary objective was to examine the effect of these differing salt sources on the nutrients NH_4-N, NO_3-N, and PO_4-P.

2.3.1 Sulfate deposition and climate change

Declining sulfate deposition and climate change has been linked to increasing concentrations of DOC in surface waters (Evans et al., 2006; Evans et al., 2005; Monteith et al., 2007). Sulfate wet deposition has declined by about 50% in Maryland since 1985. By 2014, atmospheric sulfate deposition was approximately 12 kg/ha/yr compared to approximately 24 kg/ha/yr in 1985 with sulfate ion concentrations in rainwater of 0.5 and 2.5 mg/L, respectively (NADP Program Office et al., 2015). In Galveston, sulfate deposition has not declined significantly. Wet deposition in 1985 and 2014 occurred at a rate of 10 kg/ha/yr with ion concentrations occurring in precipitation at 1.2 and 0.8 mg/L, respectively. BCS saw no substantial declines in sulfate deposition or ion concentrations. In 1985 and 2014 wet deposition occurred at a rate of 4 kg/ha/yr, while ion concentrations were approximately 0.8 mg/L.

The source of sulfate deposition was from burning of coal, particularly brown coal or lignite by coal-fired power stations. Due to the jet stream passing over the highly industrialized north-east USA, the state of Maryland would have been affected by sulfate deposition much more than Texas. If sulfate deposition was a contributing factor to DOC concentrations, it would be expected that Frederick would have higher concentrations of water extractable DOC than observed in this study. Particularly, due to some of the sample sites being present during the times of highest sulfate deposition. Yet, it was BCS, with the lowest sulfate deposition rates in 1985 and 2014, that had the highest concentrations and most significant changes in DOC over the time periods examined.

Climate change has been associated with observed DOC changes in multiple studies due to 1) increased decomposition of organic matter due to warming temperatures (Freeman et al., 2001), 2) increased atmospheric CO_2 leading to DOC enrichment due to increased plant net primary productivity (Freeman et al., 2004), or 3) increased hydrological flows due to increased rainfall causing flowpaths to shift to the organic layer where DOC is typically produced (Evans et al., 2005). However, other studies have shown that climate change alone has negligible impacts on observed DOC concentrations in the Northern Hemisphere (Evans et al., 2006). A 235 ppm increase in atmospheric CO_2 has been shown to increase DOC desorption between 14-61% in peat bog ecosystems, which researchers attributed to increased primary productivity (Freeman et al., 2004). While DOC increased an average of 91% from 1988-1993 (Evans et al., 2005), Evans et al. (2006) noted that the data from Freeman et al. (2004)

would only attribute about 1-5% of the total increase in DOC observed in the UK, as CO_2 only increased by 20 ppm between 1988 and 2006. In regards to a hydrological related influence, no consistent changes in hydrological flow regimes were observed in the UK that would explain the widespread increase in DOC (Evans et al., 2006). Therefore, climate change was not considered as a potential contributing factor in observed DOC concentrations in this study.

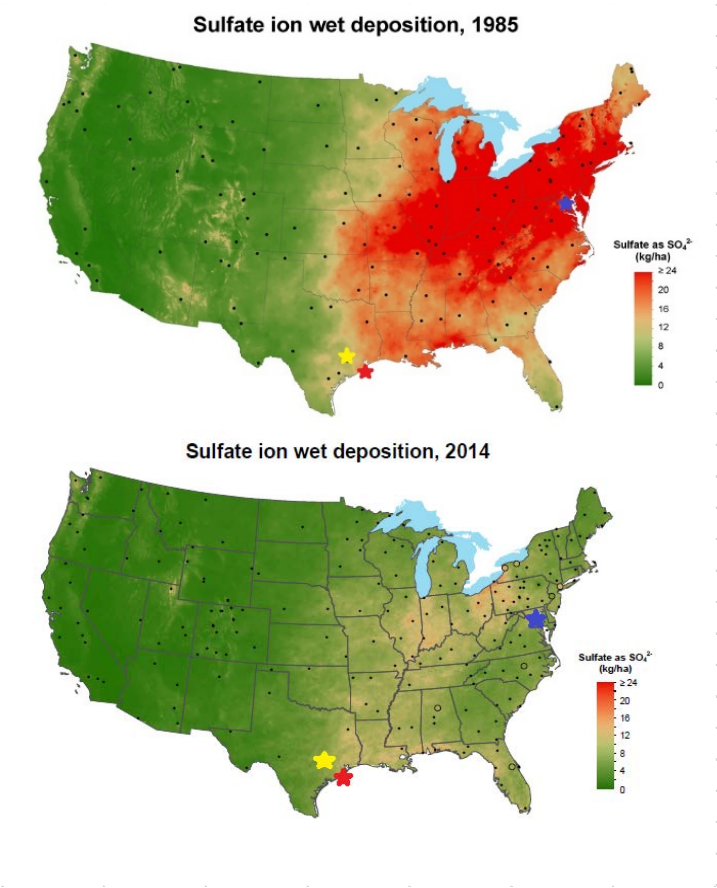

Figure 9: Sulfate deposition in the United States in 1985 versus 2014. Blue star = Frederick, Red star = Galveston and Yellow star = BCS. Modified from: (NADP Program Office et al., 2015).

2.3.2 Impact of urban sodium on DOC and DON

Several studies have examined the effect of sodium on soil organic carbon dynamics or the release of DOC and DON from soils (Green et al., 2008b; Pannkuk et al., 2011; Skene and Oades, 1995; Steele, 2011; Steele and Aitkenhead-Peterson, 2012b). Most of these studies have been conducted in the UK, Australia or Texas with the main sodium sources being deicing salts or irrigation water. In this study, estimates of water extractable DOC and DON were made using available soil variables. Water extractable DOC was estimated extremely well using independent variables Na source, pH, EC, NH_4-N, PO_4-P, DON, Mg, B, Fe, Zn and Mn. Water extractable DON was estimated equally well using independent variables sodium exposure time, sodium source, NO_3-N, PO_4-P, Mg, Na, Zn, Mn and DOC. It should be noted that exchangeable sodium percentage and sodium adsorption ratio were not selected in the backward regression analysis for water extractable DOC or DON because soil sodium concentration was already included. Inclusion of ESP and SAR would have been superfluous.

The differences in DOC's observed magnitude of response among the length of sodium exposure times is likely due to the rate of inundation with sodium. For example, in BCS, households will irrigate their lawns with highly sodic irrigation water approximately 1-2 times per day during an average 6 month growing season. In Maryland, on the other hand, lawns are only exposed to deicing salts a few months of the year. The composition of deicing salts used in Maryland was only approximately 20% NaCl until 2014 when Frederick County switched to 100% NaCl. The total tonnage used

has increased over the years, but it is spread across all roads in Frederick County's jurisdiction. In addition, these roads may not have been consistently salted since the home was built. Deicing salt records were not available before 2004. Taking soil samples from a state highway, with more consistent deicing salt application, may be more representative of the impact of NaCl on DOC concentrations.

Results for the impact of deicing salts in Frederick on DOC were surprising when compared to previous research. Green et al. (2008b) also examined the impact of deicing salt splash at varying distances on a relatively rural road in Yorkshire, UK that had been exposed to deicing salts for approximately 30 years. While this study observed no significant impact of deicing salt application, Green et al. (2008b) observed that both DOC and DON significantly increased in response to deicing salt (NaCl) application. As soil became more sodium-affected, in terms of build up over time, mobilized DOC and DON concentrations declined. They concluded at approximately 10 years of sodium exposure, readily soluble forms of DOC had already been mobilized from the soil (*"When it's gone, it's gone hypothesis").* These conflicting results could be due to several factors. Decomposition is a large source of OM to soils. Lawns are likely to be well managed and have larger sources of OM compared to Green et al. (2008b) sample sites which were subject to soil compaction from cars as well as grazing by cattle and sheep. Green et al. (2008b) samples were also collected in March, meaning it is likely that soils were more recently exposed to deicing salts than soils in Frederick, MD. Therefore, Green et al. (2008b) was potentially observing a soil's immediate response to sodium addition, while soils from this study had no sodium addition since the previous

winter season (approximately 9 months). It is possible that no change in DOC and DON was observed in Frederick soils due to equilibration of soil since the last sodium exposure. While heavily polluted soils can take from 10-40 years to recover (Vanguelova et al., 2010), taking into consideration (1) the relative infrequency of salting and (2) the composition of the deicing salts used in Frederick (only 20% NaCl until 2014) soil recovery and equilibrium may not be as long of a process. Rain events since the last salt application could have flushed DOC, DON and sodium from soil, making it appear as though deicing salts have no effect on their desorption.

Compton and Church (2011) suggested that oceanic deposition is important in decreasing DOC concentrations; however, in this study, oceanic deposition was not shown to significantly change DOC. As there have been relatively few studies regarding the impact of oceanic deposition on DOC and DON concentrations, there is little data with which to compare study results. An assumption was made that soil and/or sod for new builds were not exposed to sodium prior to installation of landscaping. Under this assumption, oceanic (NaCl) deposition does not affect DOC, DON or pH, as no significant changes in any of these analytes were observed. This could be due to the difference in soil type at the Galveston site relative to the BSC and Frederick sites and the soil in the Compton and Church (2011) study. Soils in Galveston (Entisols and Beach Material) were comprised of a high percentage of sand, while the soil analyzed in Compton and Church (2011) was an Andisol. BCS and Frederick had higher percentages of OM and clay.

Addition of sodium to an urban landscape may come in many forms; the current study examined NaCl and $NaHCO_3$. No studies were found that specifically examined the influence of NaCl versus $NaHCO_3$ on soil DOC or DON extracts. $NaHCO_3$ can neutralize the acidity of soil due to OH combining with H ions in the soil resulting in the formation of carbonates (Mirsal, 2008; Steele, 2011). Studies involving NaCl addition to soils resulted in decreased soil pH in soils not previously exposed to sodium; however, NaCl addition can increase the pH of chronically sodium exposed soil due to the displacement of H ions by Na (Green et al., 2008a; Green et al., 2008b). Surprisingly, Steele (2011) found that NaCl actually caused increased leaching of DOC from senesced vegetation. NaCl addition to soil has also been observed to inhibit C mineralization due to decreased microbial activity from increasingly acidic soils (Pathak and Rao, 1998). However, Pathak and Rao (1998) also mentioned that solubilization of organic matter at higher pH can relieve the stress of microbes in high salinity soils. This could explain why DOC exposed to $NaHCO_3$ was desorped more readily when compared to soils exposed to NaCl (Aitkenhead-Peterson et al., 2009; Green et al., 2008b). $NaHCO_3$ in the BCS soils likely increased soil and solution pH which in turn increased the solubilization of DOC, allowing microbes to withstand high salinity conditions due to higher pH levels. No definitive conclusions can be made in this study that NaCl or $NaHCO_3$ impact DOC and DON concentrations differently. It is more likely related to sodium concentrations and regularity of sodium exposure.

2.3.3 Impact of urban sodium addition on nutrients

In a study examining irrigation water sodium concentration on soil nutrients in more than 29 cities across Texas, Steele and Aitkenhead-Peterson (2012) observed that %Na had a significant positive effect on water extractable NH_4-N, and PO_4-P yet little impact on NO_3 –N. Steele and Aitkenhead-Peterson (2012) noted that increased PO_4-P concentrations may be attributed to the buildup of Na on sodium receptor sites resulting in an increasingly negative soil charge which reduced the attraction of PO_4 to soil. This would explain the negative relationship between DOC and PO_4 indicated in the model.

NH_4 was found to be significantly and positively related to Na present in the soil in Texas urban landscapes irrigated with sodic water (Steele and Aitkenhead-Peterson, 2012b). They concluded that this could be due to (1) the displacement of NH_4 by Na leading to the buildup of Na in soil and release of NH_4-N to soil solution or (2) a decrease in nitrification. This was primarily due to Na addition causing soil aggregate instability and clay dispersion and, ultimately a reduction in O_2 in pore spaces which is necessary to convert NH_4 to NO_3. DOC may also indirectly effect nitrification by increasing heterotrophic soil microbial activity, as nitrifying bacteria are generally poor competitors for NH_4 and NO_3 against heterotrophic bacteria (Bernhardt and Likens, 2002; Strauss and Lamberti, 2002). This would explain the negative relationship between DON and NO_3-N in the current study.

2.3.4 Impact of urban sodium addition on metals and metalloids

DOM has been shown to have a high binding capacity to toxic metals such as Zn, Fe, Mn, Ni, Cd and Cu (Antoniadis and Alloway, 2002; Chen et al., 2006; Stevenson,

1994). DOC and metals are known to form DOC-metal complexes. DOM-metal stability is as follows: $Cu^{2+} > Ni^{2+} + Co^{2+} > Zn^{2+} > Fe^{2+} > Mn^{2+}$ (Chen et al., 2006). pH is also an important indicator of the effectiveness of DOC binding with metals, with ideal pH being between 5-7 (Antoniadis and Alloway, 2002), and desorption from DOC occurring at higher pH (Impellitteri et al., 2002). Many metals, such as Fe, are strongly bound to DOC in low pH (Chen et al., 2006). While the metalloid B is not complexed with DOC but has a similar stron, positive relationship to increasing pH, and, therefore, desorps from soil at rates similar to DOC (Chauveheid and Denis, 2004; Elrashidi and O'Connor, 1982). BCS was the only city in this study that had significantly changing metal concentrations (Figure 7; Figure 8) which may be due to Na addition increasing pH and, thus, increasing solubility of DOC, resulting in desorption of metal from DOC complexes.

2.3.5 The differences between predictive models for DOC and DON

There were multiple differences between the predictive models for DOC and DON. It appears that those independent variables which were only chosen for DOC (pH, EC, NH_4, DON, B, Fe) were either directly or indirectly related to pH. As sodium ions increase in the soil, it replaces cations (Ca^{2+}, Mg^{2+}, H^{+}) and, ultimately, increases EC. As H^{+} increases in the soil, pH is increased which, ultimately increases DOC solubilization. The last 2 independent variables chosen for DOC and not DON were the metalloid/metal: B^{3+} and Fe^{2+}. Boron has been shown to have a positive linear relationship with DOC; while the exact mechanism is not known, this relationship is likely a combination of (1) water extractable boron is also highly correlated with pH

(Elrashidi and O'Connor, 1982) or (2) boron and DOC are both highly correlated with OM and solubilized at high pH (Chauveheid and Denis, 2004), therefore, similar patterns are observed for both. However, no studies were found that explored a relationship between DON and B. DOC has been shown to sorp to Fe, and higher concentrations of iron increase DOC sorption capacity (Chittleborough et al., 1992; Kaiser et al., 1996). As BCS had the highest concentrations of iron and DOC in any of the 3 cities, it follows the patterns followed by Chittleborough et al. (1992) and Kaiser et al. (1996). Again, no studies were found which demonstrated any relationship between DON and Fe. The only variable that was chosen for DOC that was likely not related directly to pH was NH_4. As mentioned in section 2.3.3, increasing DOC can be indirectly correlated with decreasing nitrification rates by increasing heterotrophic microbial activity, which is a stronger competitor for DOC than nitrifying bacteria, ultimately resulting in a buildup of NH_4 (Bernhardt and Likens, 2002; Strauss and Lamberti, 2002)

The independent variables that were chosen for DON and not DOC were time of exposure to sodium, NO_3, Na^+, and DOC. It's likely that the differences in DON independent variables were related to the microbial respiration rates. As soil is consistently exposed to sodium and Na^+ ions continually accumulate the respiration rates of nitrifying bacteria decline (Wong et al., 2008). This would directly prevent the microbial community from metabolizing the same quantities of DON when not exposed to sodium. Microbial communities don't directly metabolize DON, extracellular enzymes cleave amino acids from DON and microbes nitrify the amino acid into NO_3. When the amino acid is cleaved from the DON, it becomes DOC. Therefore, the effect

on microbial communities is twofold: (1) Sodium addition reducing nitrifying bacteria respiration rates and (2) increasing DOC concentrations altering microbial community composition to being dominated by heterotrophic bacteria and reducing nitrifying bacteria, further reducing nitrification.

2.4 Conclusions

Both hypothesis 1 and 2 were supported in this study. It was found that sodium exposure does affect DOC and DON. As shown in the model it directly effects DON by impacting microbial respiration rates and indirectly effects DOC by increasing pH. Length of time of exposure was also deemed crucial. As shown in BCS, DOC, DON and SAR continued to increase in response to sodium addition. While in Frederick, with low concentrations of sodium, saw little change in DOC concentrations. Finally, as represented in the model, sodium source was an important predictor of DOC and DON. It does not necessarily mean that $NaHCO_3$ has a greater impact than NaCl on DOC and DON desorption. More research would need to be done to confirm that conclusion, as Green et al. 2008b did find that NaCl had a significant effect on DOC and DON. It is likely that the sodium source represented in the model is indicative of particular characteristics of that city which could include rate of sodium addition, time since last exposure to sodium, or city soil characteristics. Soil characteristics of each site will be compared to its soil chemistry to determine the possible impacts on measured concentrations of DOC and DON.

The mechanisms involved in DOC and DON release in soils, based on our model, is not straightforward. It involves multiple variables, and while DON and DOC are often highly correlated with each other (including this study), there are many factors which are either directly or indirectly related to their measured concentrations in solution. It will be important to evaluate if this model can be applied to other soil types and locations or whether this model is site-specific. DOC and DON desorption could be influenced by different independent variables in other locations.

CHAPTER III

EFFECT OF SHAKING TIME ON THE EXTRACTION OF DOC, DON AND NUTRIENTS FROM URBAN SOILS AND CONCLUSIONS

There is no definitive extraction method for DOC and DON (Jones and Willett, 2006). Typically, DOC and DON extraction involves sieving soil and shaking with a concentrated salt solution or an ultra-pure or double deionized water (DDW) using a low soil weight to water ratio for a period of time ranging between 1 and 3 hours (Jones and Willett, 2006). Researchers generally follow some variation of these methods, but studies have shown that different shaking times, processing temperatures, soil storage procedures, extractant used and sample preparation can change the concentrations of extracted DON and DOC (Carrillo-Gonzalez et al., 2013; Guigue et al., 2014; Jones and Willett, 2006; Sharp et al., 1995; Sharp et al., 2002) as well as recovery of nutrients (Carrillo-Gonzalez et al., 2013; Christ and David, 1996). Inconsistent method procedures can either overestimate organic nutrients through release from microbial cells or underestimate them due to organic nutrient degradation during extraction (Jones and Willett, 2006). Without a consistent extraction methodology, comparison of DOC, DON and nutrient concentrations among studies could be invalid.

Jones and Willett (2006) investigated the effect of different solvents, temperatures, shaking times, varying soil:water ratios, utilizing air-dried or field moist soils, sieved versus un-sieved soil, and centrifuging prior to filtering solution. Jones and Willett (2006) conducted their study using soils from natural, undisturbed ecosystems in one geographical region. Soil shaking time, sieving soil, air drying versus field moist

soils, and seasonality had the greatest effect on altering concentrations of DON and DOC extracted from soil. DOC and DON concentrations were significantly increased when sieved to <2 mm, air dried, and shaken for longer periods of time. DOC and DON undergoes a natural seasonal flux; for example, DON recovery can vary 5-fold throughout the year (Jones and Willett, 2006), therefore, soil samples taken during different times of the year can have large variations in DOC and DON concentrations.

Another study examining the effect of tillage and cropping on extractable soil DOC, DON and nutrients found that the type of extractant used had an influence on the concentrations of DOC and DON extracted. K_2SO_4 (0.5 M) was observed to extract the most DOC (176-290 µg/g), while $CaCl_2$ (10 mM) extracted the least (49-139 µg/g) (Carrillo-Gonzalez et al., 2013). The chemical extractants $CaCl_2$ and KCl were responsible for the decoupling of DON and DOC, while cold water, hot water and K_2SO_4 retained the relationship between DON and DOC (Carrillo-Gonzalez et al., 2013). This led the researchers to conclude that DOC and DON respond differently when exposed to different chemical environments

Less research has examined the aforementioned effects on extractable soil nutrients such as NH_4-N, NO_3-N and PO_4-P. For NO_3-N, a DDW extract at a 1:5 soil:water ratio and shaken for 15 min extracted similar concentrations when compared to 10 mM $CaCl_2$, 2 M KCl and 0.5 M K_2SO_4 extracts (Carrillo-Gonzalez et al., 2013). Hot water extracts tended to extract the most NH_4-N and DON from an agricultural soil (Carrillo-Gonzalez et al., 2013). Another study suggested use of 0.01 M $CaCl_2$ as a single extraction method for NH_4, P, and NO_3N (Houba et al., 1990). Haney et al.

(2006) suggested a new extractant (H3A) that could be used to extract NO_3-N, NH_4-N and PO_4-P that would mitigate the need to use different extractants for soil NO_3-N, NH_4-N and P. The extractant was a combination of lithium citrate, citric acid, malic acid, oxalic acid, EDTA and DTPA (Haney et al., 2006). They tested the new extractant against extracts typically used such as Mehlich 3, Olsen, 1 M KCl and DDW. The soils they used had a range of clay and OM content with pH values ranging from 4.7 to 8.4. They found that when H3A was used, measurements of NO_3-N, NH_4-N, and P were consistent across soil types. A further modification to the H3A extract (H3A-2) was published in 2010 where the EDTA and DTPA were removed from the model extractant and the concentrations of lithium citrate, malic acid and oxalic acid were reduced and the concentration of citric acid increased (Haney et al., 2010). The researchers thought that H3A-2 was more accurate in estimating NO_3-N, NH_4-N, P, K, Ca and Zn and that the original extract H3A inaccurately inflated their concentrations

Choice of extractant used to determine nutrient concentrations is important. Some extractants can remove all forms of a nutrient, while others tend to only remove the plant available forms which may be a more representative measurement (Schoenau and Huang, 1991). For example, Schoenau and Huang (1991) determined that $NaHCO_3$ tended to remove more P than was plant available and was not preferable over other extraction methods such as anion exchange.

The objective of this study was to determine the effect of a 2 hour and 4 hour shaking time on water extractable DOC, DON, NH_4-N, NO_3-N and PO_4-P from urban soils from four US cities.

3.1 Methods

3.1.1 Experimental design

Soil samples were collected from four US cities exposed to different sodium sources (NaCl in the form of deicing salts or sea salt deposition and NaHCO3 in irrigation water). Frederick, MD (n=35), Bryan-College Station, TX (BCS) (n=34), Chicago, IL (n=33) and Galveston, TX (n=35) (Table 10;Table 11). To determine which homes to sample, a combination of real estate sites was used, which gave the build year and home details such as in-ground irrigation. Google Earth was also utilized to check the history of the site in terms of whether it was a new build on native land or a new build on previously residential land. In Frederick, sites were chosen by cross-referencing roads under county jurisdiction for deicing salt application. For BCS there was no information about the history of each home's irrigation system; thus, it was assumed to be present at the site since initial construction. Galveston sites were selected by obtaining a sample set representative of the length of the island.

To assess time of exposure, an assumption was made that grass was provided as sod when the home or road was constructed or, if seeded, that top soil was obtained from a location unlikely to have experienced Na^+ exposure. An equal distribution of samples was taken from age ranges of a) 0-5 years, b) 6-10 years, c) 11-20 years, d) 21-30 years and e) greater than 30 years.

Table 10: Soil characteristics of four cities examined. CEC was obtained from cations measured from 4 hour shake extracts. *Source: (Natural Resource Conservation Service and University of California Davis)*

City	Soil Group	Sample Size	Clay	Sand	Silt	OM	Ksat	BD	CEC
		%	%	%	%		mm/hr	g/cm^3	cmol charge /kg soil
Frederick	Alfisol	54.3	18±5	30±8	52±4	2±1	48±26	1.3±0.1	13±4
	Inceptisol	14.3	17±4	38±7	45±9	2±1	60±37	1.4±0.1	15±4
	Ultisol	31.4	19±2	40±5	41±6	2±0	31±7	1.4±0.1	13±2
Galveston	Entisol	74.3	5±0	94±0	1±0	1±0	331±0	1.6±0	3±0
	Beach	25.7	2±0	98±0	1±0	0±0	508±0	1.5±0	3±0
BCS	Alfisol	97.1	11±2	68±7	21±6	1+0	44±53	1.5±0.1	5±1
	Vertisol	2.9	50±0	22±0	28±0	2±0	0.8+0	1.8±0	50±0
Chicago	Urban-Entisol	100	35±0	17±0	48±0	2±0	4±0	1.9±0	20±0

Table 11: Climate characteristics of the four cities examined for this study.

Site	**Average Annual Precipitation (mm)**	**Average Annual Snowfall (mm)**	**Average Max Temperature (°C)**	**Average Min. Temperature (°C)**
Bryan-College Station, TX	1017.5	0	26.2	15.0
Galveston, TX	1093.5	0	24.7	17.2
Frederick, MD	1110.2	406.4	17.2	5.7
Chicago, IL	992.8	942.3	15.2	6.4

Source of all data: (Arguez et al., 2010)

3.1.2 Sample collection and processing

Soil samples of 15 cm depth were collected at each site using a 2 cm diameter soil probe. Soils were air dried immediately after collection and sieved using a 2 mm sieve when dry.

3.1.3 Water extraction of soils

For the 2 hour shaking time, 50 mL high density polyethelene (HDPE) centrifuge tubes were filled with 3 g of soil and 30 g ultrapure water. After 2 hr of shaking, samples were centrifuged at 19,000 g-force for 15 min at room temperature. For the 4 hour shaking time, 250 mL HPDE bottles were filled with 25 g of soil and 250 mL of ultrapure water. After 4 hr of shaking, samples were centrifuged at 19,000 g-force for 5 minutes at room temperature. The supernatant for both shake times were tested for pH and electro-conductivity (EC) and recorded. The extracts were then filtered through Whatman GF/F filters (0.7 µm nominal pore size) to remove any remaining floating organic particles prior to analysis

3.1.4 Chemical analysis

A high-temperature Platinum-catalyzed combustion with a Shimadzu TOC-V_{CSH} and Shimadzu total measuring unit (Shimadzu Corp. Houston, TX, USA) was used to measure DOC and TDN. USEPA method 415.1 was used to measure DOC by acidifying the sample and sparging for 4 min using C-free air.

NO_3-N, NH_4-N, and PO_4-P were measured using a Westco Scientific Smartchem Discrete Analyzer (Westco Scientific Instruments Inc. Brookfield, CT, USA). USEPA method 353.3 was used to measure nitrate (NO_3-N), utilizing the Cd-Cu reduction method and USEPA method 350.1 was used to measure ammonium (NH_4-N) utilizing the phenate hypochlorite method with a sodium nitroprusside enhancement. PO_4-P was quantified as orthophosphate-P using the ammonium molybdate method (USEPA 365.1). DON is estimated as the difference between TDN and the sum of NO_3-N and NH_4-N.

3.1.5 Statistical analyses

A univariate analysis of variance was used with each nutrient in turn as the dependent variable, and city and shake time as the independent variables to ascertain if there was a significant effect of city (differing soil classes and sodium sources), shaking time, or an interaction between city and shaking time. If shake time was significant ($P<.05$), a Fisher's Least Significant Difference (LSD) test was conducted on each nutrient.

3.2 Results

Shake times were not always significantly different for analytes among cities. There was no significant difference between shake times for any of the extracted soil analytes in Frederick, while Galveston and Chicago had 6 and 4 analytes with significant differences, respectively (Figure 10). In BCS, only pH and DON were significantly impacted by differences in shake time. All parameters were significantly affected by shake time in at least 1 city, with the exception of NO_3-N (Figure 10).

pH was most significantly impacted by shake time across cities; there was a significant difference in Chicago, Galveston and BCS (Figure 10;Table 12). In all cities, pH decreased with increasing shake time. EC increased significantly in Chicago and Galveston. Ammonium increased with increasing shake time, but the change was only significantly different in Chicago and Galveston. Phosphate only changed significantly with shake time in Chicago, where it increased with a four hour shake time when compared to two hour shake time. TDN increased in all cities but response to shake time

was significant in only Galveston. Surprisingly, DOC only changed significantly with shake time in Galveston, though it also increased slightly in BCS and Chicago. Frederick DOC concentrations remained the same despite shake time. DON responded more often to changing shake time than DOC. DON increased significantly in Galveston and BCS with longer shake times. In Frederick and Chicago extracted concentrations of DON remained the same.

Ultimately, there were no consistent trends in response to shake time. Generally, increasing shake time tends to increase concentrations slightly, though the change often does not end up being significant. Only pH consistently declined in response to shake time in all cities. The only other analyte which declined in response to shake time was EC in BCS; however, the decrease was insignificant.

Table 12: P-values associated with LSD test (α=.05). Numbers in bold indicate a significant difference between shake times.

Testing Parameter	P-Value			
	Chicago	**Galveston**	**Frederick**	**BCS**
pH	**0.00**	**0.00**	0.10	**0.00**
EC	**0.04**	**0.00**	0.26	0.70
NH_4-N	**0.00**	**0.05**	0.11	0.69
NO_3-N	0.39	0.91	0.71	0.96
PO_4-P	**0.00**	0.09	0.38	0.46
DOC	0.32	**0.02**	0.32	0.05
TDN	0.14	**0.04**	0.56	0.05
DON	0.83	**0.00**	0.82	**0.00**

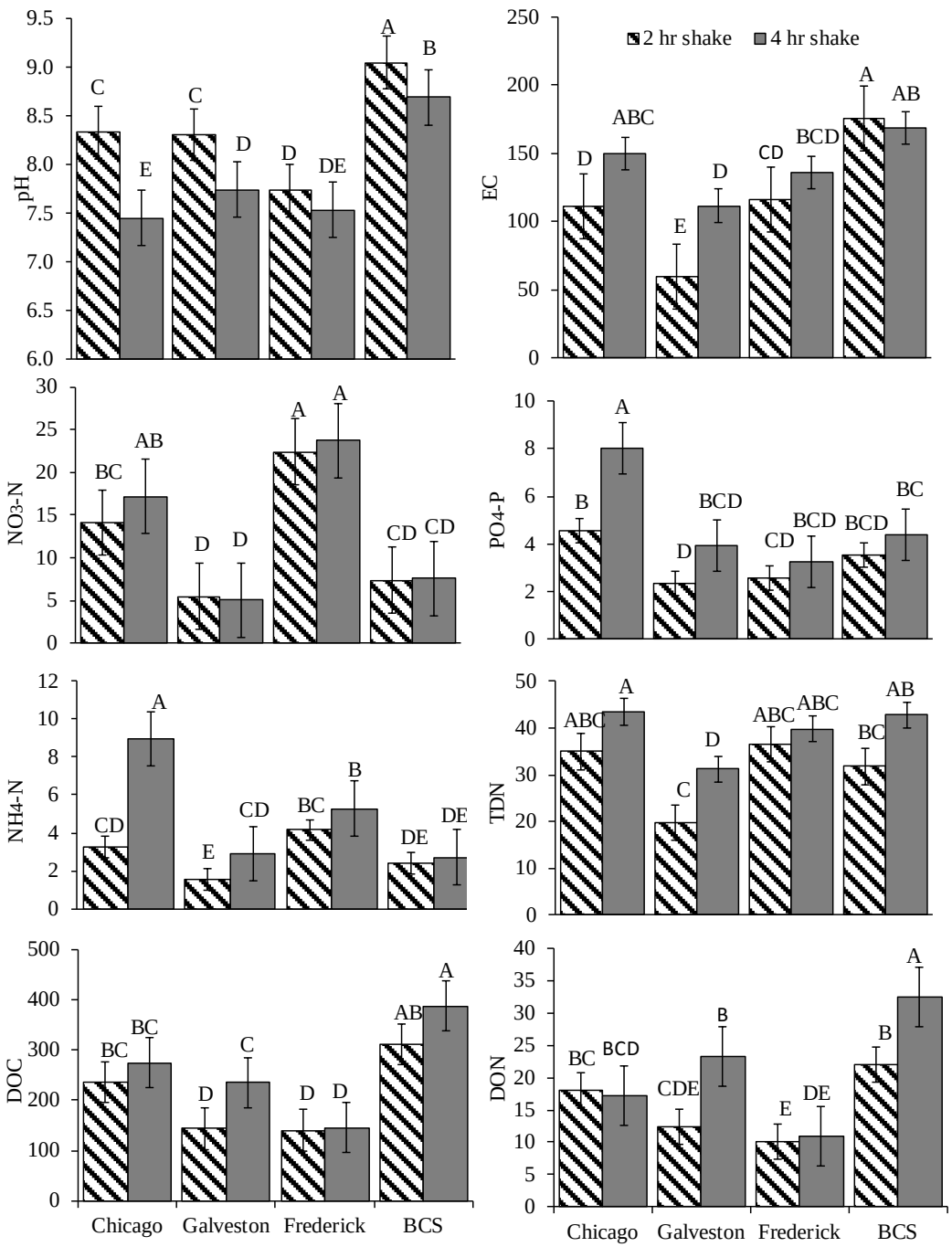

Figure 10: Mean pH, EC, and water extractable NO_3-N, NH_4-N, PO_4-P, TDN, DOC, and DON measured comparing 2 and 4 hour shake time. Error bars represent standard error. Columns with the same letter are not significantly different.

3.3 Discussion

This study examined the effect of shaking time on water extractable DOC, DON and nutrients in the soils of four US cities. Several studies have examined the length of shaking time on the mass of DOC and DON extracted from soil (Guigue et al., 2014; Jones and Willett, 2006; Ros et al., 2009). Other studies have examined the length of shaking time on inorganic nutrients such as phosphorus, (Fuhrman et al., 2005; Kleinman et al., 2002; Schoenau and Huang, 1991) as a means to find the best method for determining plant available P. Knowing the effect of shaking time on nutrient and organic matter concentrations can provide a more efficient means of comparison across studies.

3.3.1 Impact of shake time on DOC and DON

Positive relationships have been reported for water extractable DOC and DON and the amount of time they were shaken prior to extraction (Jones and Willett, 2006). This suggests that longer shaking times would increase the amount of DOC and DON recovered from soil. While DOC and DON did increase in response to shake time, there was, surprisingly, no significant increase except in Galveston (Figure 10). This contradicts the findings by Jones and Willett (2006), which found a positive relationship between DOC and DON concentrations and shake time. Jones and Willett's (2006) study was representative of soils in natural undisturbed ecosystems, while our soils were located in urban environments.

Soil types can vary significantly across regions. Ros et al. (2009) found that soil chemistry is influential in rates of N mineralization and amount of DON extracted from

soil solution which supported the findings of Carrillo-Gonzalez et al. (2014) for DOC. The amount of DON extracted using different extractants in the Ros et al. (2009) study was as follows: $NaHCO_3$>$CaCl_2$>K_2SO_4. However, Carrillo-Gonzalez et al. (2009) found 10 mM $CaCl_2$ extracted less DOC (49-139 µg/g) than 0.5 M K_2SO_4 (176-290 µg/g). Jones and Willett (2006) found that 2 M KCl and 0.5 M K_2SO_4 did not have an effect on DOC extraction. These contrasting results could be due to the different soil types used. For example, Ros et al. (2009) observed that extractable organic nitrogen was 40-50% higher in clay soils compared to sandy soils. As there were no consistent results of 2 and 4 hour shakes on the analytes throughout cities, it is likely that the cities' soil characteristics (soil type, % OM and antecedent soil chemistry in terms of sodium) may have been the determining factor in whether a significant difference between 2 and 4 hour shaking times was observed.

No research has been conducted that examined differing extraction methodology on urban soils. All research examining extraction methodologies have used natural, undisturbed soils or agricultural soils (Carrillo-Gonzalez et al., 2013; Jones and Willett, 2006; Ros et al., 2009). In this study, all soil samples were taken from urban environments with potentially enhanced sodium exposure. While changing shaking times may have a different effect on undisturbed and urban soils, excess sodium exposure does not appear to influence whether shaking time can significantly impact measured DOC and DON concentrations. For example, the cities of Frederick and Chicago had the same source of sodium exposure (deicing salts) yet Chicago observed significant differences in 4 analytes (pH, EC, NH_4-N and PO_4-P) while Frederick

showed no effect of shaking time on any of the analytes examined. If sodium source and its concomitant effect on soil chemistry was a factor in significant shaking time differences, then these two cities would likely have had similar results. Because DOC and DON did not respond consistently to sodium source or shaking time, the shaking time may be more influenced by the different soil environmental chemistry in Frederick, Galveston and BCS. Jones and Willett (2006), Carrillo-Gonzalez et al. (2013), and Ros et al. (2009) observed different responses of DON and DON to chemical extractants. Because DOC and DON responded differently to chemical extractants, they would likely have contrasting responses to various soil chemical environments. BCS and Frederick both had the fewest analytes affected by shaking time. Analyte concentrations were also different between BCS and Frederick, with the exception of TDN and PO_4-P (Figure 10), climate and average annual precipitation volumes also differed among cities (Table 11). The only similarity among the cities that was apparent in the collected data was major soil type. Alfisol was the major soil order in BCS and Frederick, while Entisol was the major soil order in Chicago and Galveston. In an effort to determine why some of the city soils showed significant increases in extracted DOC, DON and nutrients while others did not, a univariate analysis of variance was conducted on soil CEC. While there was no significant difference in soil CEC among the cities, the soil CEC for Frederick had the highest value, followed by BCS, Chicago and Galveston, respectively. The values for soil CEC had an inverse relationship with the number of analytes affected by shaking time. For example, Frederick had the least amount of analytes affected by shake time, followed by BCS, Chicago and Galveston, respectively. CEC is often indicative of

higher OM and clay in soil, which generally have higher adsorption capacities (Department of Crop and Soil Sciences, 2007). CEC was lowest in Galveston, which is typical in sandy soils (Department of Crop and Soil Sciences, 2007), and also had low percentages of OM and clay. Sandy soils are also generally composed of SiO_2 which has a neutral charge, allowing for increased desorption of DOC, DON and nutrients when compared to clay soils. While this is not a definitive relationship, it may be indicative of the importance of shake time's influence on different soil types. More research should be conducted on the impact of varying methodologies on different soils.

3.3.2 Impact of shaking time on inorganic N and P

Many studies have shown that extractable soil nitrate is affected by air drying the soils resulting in a large flush of nitrate compared to extracting field moist soils (Harada and Hayashi, 1967; Mian et al., 2008). The air drying may have induced a large flush of nitrate-N which may explain why it did not show any significant difference in nitrate-N concentrations between the two shaking times. Mian et al. (2008) hypothesized that the rapid flush of nitrate was likely due to (1) nitrate produced and stored during the drying process or (2) microbial cell lysis. Researchers explained that osmotic shock to microbial cells during the rewetting process initiates the release of intracellular cell solute after cell lysis (Mian et al., 2008). That would explain the relatively short lived nitrate flush, however, it doesn't account for the concentrations seen in some studies since most of the intracellular solutes are composed of carbon and amino acids (Halverson et al., 2000). The most plausible explanation, according to Mian et al. (2008), may be that nitrate

produced during the drying process was not taken up by plants and thus stored in soil, causing a rapid flush when rewetted.

Mian et al (2008) also studied the impact of rewetting dry soil on soil ammonium recovery and found that its response to rewetting was more subtle. While there was a slight initial increase in ammonium that increase was not sustained, and ammonium concentrations remained steady throughout their experiment. This is indicative of what was seen in our results in BCS and Frederick, but does not explain the significantly higher concentrations observed for a 4 hour shaking time compared to a 2 hour shaking of Chicago and Galveston soils. Soils with a lower buffering capacity, such as sandy soils, do not retain ammonium as well as clayey soils, which have higher buffering capacities (Wang and Alva, 2000). This could potentially explain why Galveston saw a significant increase in ammonium-N after being shaken for 4 hours. Other studies have attributed freeze-thaw conditions to high nitrogen mineralization rates and the release of ammonium from organic and inorganic colloids, ultimately leading to increased ammonium concentrations (Freppaz et al., 2007; Stanford and Smith, 1972).

Freeze thaw cycles have also been attributed to the increase of dissolved organic phosphorus (DOP) and total dissolved phosphorus (TDP) (Freppaz et al., 2007). Chicago was also the only city in which an increase in phosphate-P concentration occurred with a four hour shaking time, consistent with the ammonium findings. When samples were collected in Chicago, soils were frozen and covered with a layer of snow.

3.3.3 Conclusion

No consistent effect of shake time was observed for pH, EC, DON, DOC, TDN, PO_4-P, and NH_4-N in any city. Only NO_3-N had no significant difference between shake time in any city. Therefore, it is likely shake time, in and of itself, is less important than the characteristics of the study site being sampled. Sodium input also seemed to have little significance when determining the impact of 2 and 4 hour shaking times. Researchers should look at the characteristics of the soil type, as well as climate, to determine what shake time is necessary. While Jones and Willett (2006) suggested a consistent methodology for the extraction of DOC, it is unlikely widely applicable. This study shows the variety of results obtained by just changing shake time in 4 cities throughout the United States. Therefore, extraction method is probably best determined by analyzing the characteristics of sample sites rather than applying one general extraction methodology.

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APPENDIX A

Table 13: Physical and chemical soil attributes for the soils collected in Frederick, MD

Lat	Long	Series Code	Soil Series	Group	Clay	Sand	Silt	OM	Ksat	BD	CEC
						%			mm/hr	g/cm^3	cmol charge /kg soil
39.3282	77.3491	LyC	Linganore	Alfisol	19.0	34.0	47.0	2.0	32.4	1.06	10.2
39.3374	77.3404	HyD	Hyattswtown	Alfisol	20.0	26.5	53.5	1.3	32.4	1.59	10.8
39.3431	77.3258	GmB	Gleneig	Ultisol	20.0	42.1	37.9	2.0	33.0	1.31	12.6
39.3740	77.3535	CeB	Catoctin	Inceptisol	12.5	45.7	41.8	1.3	100.8	1.45	11.0
39.3818	77.3586	BkD	Brinklow	Ultisol	17.0	31.5	51.5	2.5	9.7	1.36	15.0
39.4071	77.3321	had	Hatboro	Inceptisol	15.0	30.1	54.9	3.0	33.0	1.36	22.0
39.4109	77.3384	GoC	Glenville	Ultisol	15.0	30.1	54.9	2.5	32.4	1.36	8.2
39.4072	77.3586	WrB	Whiteford	Ultisol	20.0	42.1	37.9	2.0	32.4	1.58	14.2
39.4233	77.3920	BtB	Buckeystown	Alfisol	8.5	48.8	42.7	2.0	33.1	1.36	8.2
39.4451	77.2808	BhE	Blocktown	Ultisol	20.0	42.1	37.9	1.3	32.4	1.59	11.0
39.4592	77.4116	DwB	Duffield	Alfisol	22.5	22.4	55.1	3.0	32.4	1.32	17.5
39.4617	77.4244	DwB	Duffield	Alfisol	22.5	22.4	55.1	3.0	32.4	1.32	17.5
39.4492	77.4406	HcB	Hagerstown	Alfisol	28.0	18.4	53.6	3.0	82.8	1.37	22.5
39.4798	77.4574	TrB	Trego	Ultisol	15.0	44.3	40.7	3.0	32.4	1.42	15.0
39.4387	77.5028	HgC	Highfield	Alfisol	15.0	30.1	54.9	2.0	32.4	1.37	11.6
39.4306	77.4744	MeD	Mt. Airy	Inceptisol	20.5	42.0	37.5	2.0	32.4	1.39	14.4
39.3879	77.4511	PrB	Penn	Alfisol	19.0	26.0	55.0	2.0	82.8	1.36	13.7
39.3862	77.4318	DwB	Duffield	Alfisol	22.5	22.4	55.1	3.0	32.4	1.32	17.5
39.3719	77.4444	AfB	Adamstown	Alfisol	20.0	26.5	53.5	1.5	32.4	1.26	10.8
39.3664	77.4407	DtB	Duffield	Alfisol	22.5	22.4	55.1	3.0	32.4	1.32	17.5
39.3063	77.4812	DtB	Duffield	Alfisol	22.5	22.4	55.1	3.0	32.4	1.32	17.5

39.3611	77.5550	MvC	Myersville	Alfisol	15.0	35.0	50.0	2.0	100.8	1.42	11.6
39.3677	77.4460	BtB	Buckeystown	Alfisol	8.5	48.8	42.7	2.0	33.1	1.36	8.2
39.3880	77.4723	KnC	Klinesville	Inceptisol	15.0	30.1	54.9	0.4	100.8	1.65	11.6
39.4567	77.4357	PqB	Reaville	Alfisol	18.0	29.0	53.0	2.5	33.0	1.36	15.0
39.4739	77.4619	GgB	Gleneig	Ultisol	20.0	42.1	37.9	2.0	33.0	1.31	12.6
39.4912	77.4143	PeB	Penn	Alfisol	19.0	26.0	55.0	2.0	82.8	1.36	13.7
39.4044	77.3392	MeC	Mt. Airy	Inceptisol	20.5	42.0	37.5	2.0	32.4	1.39	14.4
39.4111	77.3395	GmB	Gleneig	Ultisol	20.0	42.1	37.9	2.0	33.0	1.31	12.6
39.3408	77.3337	GmB	Gleneig	Ultisol	20.0	42.1	37.9	2.0	33.0	1.31	12.6
39.3367	77.3387	GmB	Gleneig	Ultisol	20.0	42.1	37.9	2.0	33.0	1.31	12.6
39.3315	77.3582	MvB	Myersville	Alfisol	15.0	35.0	50.0	2.0	100.8	1.42	11.6
39.3348	77.3203	GgB	Gleneig	Ultisol	20.0	42.1	37.9	2.0	33.0	1.31	12.6
39.3289	77.2736	LyC	Linganore	Alfisol	19.0	34.0	47.0	2.0	32.4	1.06	10.2
39.3315	77.2726	LyC	Linganore	Alfisol	19.0	34.0	47.0	2.0	32.4	1.06	10.2

Table 14: Physical and chemical soil attributes for the soils collected in Galveston, TX

Lat	Long	Series Code	Soil Series	Group	Clay	Sand	Silt	OM	Ksat	BD	CEC
						%			mm/hr	g/cm^3	cmol charge /kg soil
29.2833	-94.8698	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.2937	-94.8639	Gd	Galveston	Entisol	5.0	94.4	0.6	0.3	331.2	1.67	2.5
29.2902	-94.8182	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.3200	-94.7713	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.3178	-94.7712	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.3156	-94.7690	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.3231	-94.7429	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.3196	-94.7487	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.3185	-94.7503	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2778	-94.8123	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.2792	-94.8247	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.2771	-94.8473	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.2685	-94.8758	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2631	-94.8712	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2524	-94.8701	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2259	-94.9185	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2197	-94.9090	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2095	-94.9286	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2067	-94.9328	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2059	-94.9365	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2035	-94.9396	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.2032	-94.9415	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1985	-94.9461	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3

29.1979	-94.9469	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1990	-94.9474	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1998	-94.9865	Mu	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1431	-95.0453	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1409	-95.0514	Mu	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1391	-95.0472	GaB	Galveston	Entisol	5.0	94.4	0.6	0.3	331.2	1.67	2.5
29.1353	-95.0485	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1275	-95.0600	BBBX	Beaches	NA	1.5	97.9	0.6	0.0	507.6	1.5	3
29.1115	-95.0852	Gc	Galveston	Entisol	5.0	94.4	0.6	0.3	331.2	1.67	2.5
29.1120	-95.0874	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1117	-95.0872	Mt	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3
29.1364	-95.0492	Mn	Mustang	Entisol	5.0	94.4	0.6	0.6	331.2	1.57	3

Table 15: Physical and chemical soil attributes for the soils collected in Chicago, IL

Lat	Long	Series	Name	Group	Clay	Sand	Silt	OM	Ksat	BD	CEC
					%				mm/hr	g/cm^3	cmol charge /kg soil
41.9736	-87.7593	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9732	-87.7580	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9704	-87.7557	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9714	-87.7425	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9740	-87.7242	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9830	-87.7389	2811A	Urban-Alfic-Udarent	Entisol	36.0	8.0	56.0	2.5	3.3	1.85	19.4
41.9855	-87.7380	2811A	Urban-Alfic-Udarent	Entisol	36.0	8.0	56.0	2.5	3.3	1.85	19.4
41.9887	-87.7364	533	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9956	-87.7377	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9958	-87.7370	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9729	-87.6980	2800A	Urban-Psamment	Entisol	25.0	35.0	40.0	2.0	10.2	1.78	18.3
41.9722	-87.6982	2800A	Urban-Psamment	Entisol	25.0	35.0	40.0	2.0	10.2	1.78	18.3
41.9518	-87.7181	392A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9509	-87.7370	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4

41.9473	-87.7327	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9436	-87.7450	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9566	-87.7454	533	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9664	-87.7809	2811A	Urban-Alfic-Udarent	Entisol	36.0	8.0	56.0	2.5	3.3	1.85	19.4
41.9643	-87.7820	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9615	-87.7883	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9474	-87.7957	2811A	Urban-Alfic-Udarent	Entisol	36.0	8.0	56.0	2.5	3.3	1.85	19.4
41.9331	-87.8062	533	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9405	-87.8139	2811A	Urban-Alfic-Udarent	Entisol	36.0	8.0	56.0	2.5	3.3	1.85	19.4
41.9550	-87.8107	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9663	-87.8087	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9695	-87.8063	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9737	-87.8134	2811A	Urban-Alfic-Udarent	Entisol	36.0	8.0	56.0	2.5	3.3	1.85	19.4
41.9687	-87.8376	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4
41.9790	-87.8215	2811A	Urban-Alfic-Udarent	Entisol	36.0	8.0	56.0	2.5	3.3	1.85	19.4
41.9803	-87.8215	2811A	Urban-Alfic-Udarent	Entisol	36.0	8.0	56.0	2.5	3.3	1.85	19.4
41.9816	-87.7942	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4

41.9478	-87.8702	534A	Urban-Orthent	Entisol	44.0	8.0	48.0	1.3	1.0	1.98	23.4	

Table 16: Physical and chemical soil attributes for the soils collected in Bryan/College Station, TX

Lat	Long	Series	Name	Group	Clay	Sand	Silt	OM	Ksat	BD	CEC
						%			mm/hr	g/cm^3	cmol charge /kg soil
30.5975	96.3303	BrB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.5878	96.3028	TuA	Tabor	Alfisol	14.0	69.6	16.4	0.8	32.4	1.63	3.5
30.5830	96.3033	TuA	Tabor	Alfisol	14.0	69.6	16.4	0.8	32.4	1.63	3.5
30.5658	96.2894	BoA	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.5651	96.2905	BuB	Burleson	Vertisol	50.0	22.1	27.9	2.0	0.8	1.78	50
30.5652	96.2888	BoA	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.5647	96.2890	BoA	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.5605	96.2851	SnB	Singleton	Alfisol	12.5	70.9	16.6	0.8	32.4	1.62	6
30.5611	96.2853	SnB	Singleton	Alfisol	12.5	70.9	16.6	0.8	32.4	1.62	6
30.5449	96.2879	MaA	Mabank	Alfisol	17.5	43.0	39.5	1.5	32.4	1.65	7.5
30.5436	96.2894	MaA	Mabank	Alfisol	17.5	43.0	39.5	1.5	32.4	1.65	7.5
30.5263	96.2497	BwC	Burlewash	Alfisol	10.0	65.0	25.0	1.3	32.4	1.55	4.3
30.5546	96.2306	BwC	Burlewash	Alfisol	10.0	65.0	25.0	1.3	32.4	1.55	4.3
30.5620	96.2450	ReC	Rehburg	Alfisol	7.0	83.5	9.5	0.8	331.2	1.52	3.5
30.5614	96.2487	SkB	Shiro	Alfisol	8.5	82.2	9.3	0.8	100.8	1.52	4.5
30.5626	96.2502	BwC	Burlewash	Alfisol	10.0	65.0	25.0	1.3	32.4	1.55	4.3
30.5896	96.2834	TuA	Tabor	Alfisol	14.0	69.6	16.4	0.8	32.4	1.63	3.5
30.5909	96.2837	TuA	Tabor	Alfisol	14.0	69.6	16.4	0.8	32.4	1.63	3.5
30.5905	96.2787	TuA	Tabor	Alfisol	14.0	69.6	16.4	0.8	32.4	1.63	3.5
30.5901	96.2795	TuA	Tabor	Alfisol	14.0	69.6	16.4	0.8	32.4	1.63	3.5
30.6179	96.2846	BrB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6208	96.2885	BrB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6239	96.3026	Ur-BrB	Urban	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6

30.6310	96.3167	ZcB	Zack	Alfisol	11.0	67.7	21.3	0.7	32.4	1.29	7.5
30.6286	96.3198	BrB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6684	96.3415	ZcB	Zack	Alfisol	11.0	67.7	21.3	0.7	32.4	1.29	7.5
30.6767	96.3310	BoB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6788	96.3336	BoB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6761	96.3329	ZcD	Zack	Alfisol	11.0	67.7	21.3	0.7	32.4	1.29	7.5
30.6874	96.3305	BoB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6868	96.3308	BoB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6811	96.3224	BoB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6811	96.3224	BoB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6
30.6727	96.2944	BoB	Boonville	Alfisol	10.0	68.5	21.5	0.8	32.4	1.49	6

APPENDIX B

Table 17. Raw data for 4 hour shaken water extracts

NAWA ID	City	Age	Age Class	Na Source	pH	EC µS/cm	NO_3-N	NH_4-N	PO_4-P	DOC mg/kg	TDN	DON	HCO_3
S06887	Chicago	6	6 to 10	Deicing1	7.7	135	7	4	1	136	19	8	372
S06888	Chicago	21	21 to 30	Deicing1	7.3	199	56	12	5	273	85	16	285
S06889	Chicago	30	> 30	Deicing1	7.9	120	11	14	8	470	61	36	294
S06890	Chicago	7	6 to 10	Deicing1	7.4	127	20	8	4	237	45	18	378
S06891	Chicago	91	> 30	Deicing1	7.4	114	27	8	7	232	50	15	0
S06892	Chicago	72	> 30	Deicing1	7.8	229	28	12	7	282	55	15	0
S06893	Chicago	10	6 to 10	Deicing1	7.6	229	14	14	8	569	55	27	651
S06894	Chicago	0	0 to 5	Deicing1	7.2	122	3	4	4	120	9	1	452
S06895	Chicago	24	21 to 30	Deicing1	7.6	140	5	6	6	269	31	20	377
S06896	Chicago	14	11 to 20	Deicing1	7.7	100	10	22	7	449	61	30	167
S06897	Chicago	61	> 30	Deicing1	7.6	101	24	10	5	240	49	14	280
S06899	Chicago	92	> 30	Deicing1	7.2	143	23	8	4	114	35	4	458
S06900	Chicago	3	0 to 5	Deicing1	7.5	142	8	8	5	195	28	11	517
S06901	Chicago	11	11 to 20	Deicing1	7.1	108	16	5	5	142	27	7	421
S06902	Chicago	0	0 to 5	Deicing1	7.0	154	5	2	1	146	16	9	674
S06903	Chicago	3	0 to 5	Deicing1	7.3	172	27	8	10	155	42	7	584
S06904	Chicago	21	21 to 30	Deicing1	7.4	186	16	6	49	287	38	16	583
S06905	Chicago	12	11 to 20	Deicing1	7.6	167	26	13	10	252	58	19	325
S06906	Chicago	0	0 to 5	Deicing1	7.3	134	6	5	4	126	13	2	251
S06907	Chicago	7	6 to 10	Deicing1	7.9	107	28	4	5	183	44	12	240
S06908	Chicago	27	21 to 30	Deicing1	7.9	197	10	13	11	928	95	72	185
S06909	Chicago	12	11 to 20	Deicing1	7.0	166	9	11	13	229	27	7	683
S06910	Chicago	0	0 to 5	Deicing1	7.5	141	10	5	3	194	27	12	519
S06911	Chicago	18	11 to 20	Deicing1	7.4	139	51	5	6	168	66	9	292
S06912	Chicago	59	> 30	Deicing1	7.6	110	16	12	13	242	37	9	128
S06913	Chicago	55	> 30	Deicing1	7.8	170	8	12	9	510	48	28	579
S06914	Chicago	69	> 30	Deicing1	7.4	162	15	10	12	332	47	22	309
S06915	Chicago	9	6 to 10	Deicing1	7.3	135	16	8	3	205	36	12	433

S06916	Chicago	0	0 to 5	Deicing1	6.9	138	18	9	5	142	33	6	533
S06917	Chicago	8	6 to 10	Deicing1	7.6	131	8	11	5	248	38	19	620
S06918	Chicago	15	11 to 20	Deicing1	7.1	165	12	7	14	173	29	10	597
S06919	Chicago	10	6 to 10	Deicing1	7.5	128	10	9	2	152	29	9	170
S06920	Chicago	29	21 to 30	Deicing1	7.7	220	21	12	12	671	100	67	309
S06956	BCS	13	11 to 20	Irrigation	9.1	163	10	2	3	359	40	28	824
S06957	BCS	13	11 to 20	Irrigation	9.3	170	6	2	3	403	38	30	871
S06958	BCS	60	> 30	Irrigation	8.4	78	10	2	2	250	33	20	369
S06959	BCS	56	> 30	Irrigation	7.6	60	10	2	4	231	30	18	250
S06960	BCS	30	> 30	Irrigation	9.6	250	14	4	9	949	97	79	1124
S06961	BCS	0	0 to 5	Irrigation	8.3	83	3	2	2	157	18	14	407
S06962	BCS	28	21 to 30	Irrigation	9.3	122	7	3	5	318	42	32	591
S06963	BCS	60	> 30	Irrigation	8.1	45	7	2	14	273	31	22	0
S06964	BCS	8	6 to 10	Irrigation	8.4	130	5	1	1	185	20	14	635
S06965	BCS	13	11 to 20	Irrigation	9.7	402	14	8	11	1211	133	111	804
S06966	BCS	9	6 to 10	Irrigation	8.5	165	7	2	10	284	31	22	733
S06967	BCS	0	0 to 5	Irrigation	8.5	102	5	1	1	119	15	8	528
S06968	BCS	34	> 30	Irrigation	8.3	60	6	2	3	180	22	14	207
S06969	BCS	9	6 to 10	Irrigation	8.6	164	6	2	4	293	33	25	580
S06970	BCS	28	21 to 30	Irrigation	8.8	130	9	3	6	276	43	32	570
S06971	BCS	0	0 to 5	Irrigation	6.7	111	4	1	1	81	9	4	25
S06972	BCS	27	21 to 30	Irrigation	8.9	155	7	2	3	476	56	46	631
S06973	BCS	0	0 to 5	Irrigation	6.7	198	3	7	0	110	17	8	429
S06974	BCS	23	21 to 30	Irrigation	9.6	301	12	3	7	630	75	59	762
S06975	BCS	0	0 to 5	Irrigation	8.7	196	1	1	1	176	14	12	730
S06976	BCS	22	21 to 30	Irrigation	9.5	169	6	3	4	395	51	42	656
S06977	BCS	15	11 to 20	Irrigation	9.2	170	6	1	4	357	39	32	628
S06978	BCS	8	6 to 10	Irrigation	7.3	394	4	1	0	124	14	9	370
S06979	BCS	8	6 to 10	Irrigation	8.4	97	7	3	5	171	25	16	421
S06980	BCS	19	11 to 20	Irrigation	8.5	117	5	1	4	155	18	12	423
S06981	BCS	8	6 to 10	Irrigation	9.7	280	14	7	7	842	91	71	733
S06982	BCS	34	> 30	Irrigation	9.8	352	13	5	8	1158	117	99	849
S06983	BCS	23	21 to 30	Irrigation	9.7	333	14	5	6	755	89	70	1043

S06984	BCS	11	11 to 20	Irrigation	8.3	57	7	2	3	268	30	21	167
S06985	BCS	34	> 30	Irrigation	9.1	167	4	1	4	250	21	15	937
S06986	BCS	9	6 to 10	Irrigation	9.4	220	7	2	5	370	37	28	1283
S06987	BCS	0	0 to 5	Irrigation	8.5	125	4	1	1	176	13	9	567
S06988	BCS	27	21 to 30	Irrigation	8.7	96	18	7	6	1003	94	68	595
S06989	BCS	16	11 to 20	Irrigation	8.3	76	4	2	1	198	19	13	356
S06990	Galveston	30	> 30	Seasalt	7.1	106	7	6	4	283	40	27	20
S06991	Galveston	3	0 to 5	Seasalt	7.1	112	7	2	1	357	44	34	363
S06992	Galveston	7	6 to 10	Seasalt	7.0	143	6	6	10	409	51	39	48
S06993	Galveston	55	> 30	Seasalt	6.9	155	6	5	4	337	43	33	395
S06994	Galveston	50	> 30	Seasalt	7.4	130	10	10	4	245	45	25	127
S06995	Galveston	45	> 30	Seasalt	6.9	178	11	5	4	415	58	42	167
S06996	Galveston	6	6 to 10	Seasalt	7.4	128	7	4	6	280	37	27	0
S06997	Galveston	0	0 to 5	Seasalt	8.0	106	4	4	5	110	30	22	94
S06998	Galveston	7	6 to 10	Seasalt	7.8	85	2	3	2	145	18	12	98
S06999	Galveston	89	> 30	Seasalt	9.5	177	4	10	13	597	71	57	0
S07000	Galveston	27	21 to 30	Seasalt	7.0	130	7	4	11	414	49	38	0
S07001	Galveston	12	11 to 20	Seasalt	7.1	114	6	4	3	295	41	31	114
S07002	Galveston	22	21 to 30	Seasalt	8.2	103	6	3	4	446	49	39	503
S07003	Galveston	2	0 to 5	Seasalt	8.2	77	6	2	4	256	30	22	0
S07004	Galveston	34	> 30	Seasalt	7.6	60	4	3	9	347	37	30	72
S07005	Galveston	15	11 to 20	Seasalt	8.1	68	7	2	5	318	39	30	19
S07006	Galveston	10	6 to 10	Seasalt	8.2	61	4	4	5	233	28	21	41
S07007	Galveston	18	11 to 20	Seasalt	7.6	77	8	2	4	342	41	30	4
S07008	Galveston	13	11 to 20	Seasalt	8.4	101	5	2	2	176	26	19	27
S07009	Galveston	25	21 to 30	Seasalt	8.1	59	3	2	2	128	18	13	111
S07010	Galveston	11	11 to 20	Seasalt	7.6	62	3	2	1	152	20	16	114
S07011	Galveston	28	21 to 30	Seasalt	8.3	71	4	1	2	166	24	18	0
S07012	Galveston	16	11 to 20	Seasalt	8.3	93	4	2	5	195	28	22	120
S07013	Galveston	5	0 to 5	Seasalt	8.1	60	4	2	5	164	22	16	0
S07014	Galveston	23	21 to 30	Seasalt	8.3	63	3	2	2	142	18	14	51
S07015	Galveston	10	6 to 10	Seasalt	8.0	103	11	2	1	126	25	13	78
S07016	Galveston	8	6 to 10	Seasalt	7.6	62	2	0	1	109	13	11	50

S07017	Galveston	21	21 to 30	Seasalt	7.6	56	4	1	3	124	19	15	185
S07018	Galveston	4	0 to 5	Seasalt	8.5	97	1	1	2	124	13	11	44
S07019	Galveston	35	> 30	Seasalt	7.5	76	4	1	6	141	21	15	5
S07020	Galveston	31	> 30	Seasalt	8.3	78	5	1	2	107	19	13	138
S07021	Galveston	14	11 to 20	Seasalt	8.3	21	6	1	1	151	23	17	41
S07022	Galveston	0	0 to 5	Seasalt	8.3	129	1	1	2	114	11	9	385
S07023	Galveston	9	6 to 10	Seasalt	8.2	86	4	2	2	181	25	19	288
S07024	Galveston	0	0 to 5	Seasalt	7.6	56	3	1	2	103	16	12	83
S06921	Frederick	26	21 to 30	Deicing2	6.9	121	8	2	1	107	17	6	473
S06922	Frederick	7	6 to 10	Deicing2	7.2	113	7	5	1	141	22	11	477
S06923	Frederick	20	11 to 20	Deicing2	7.1	127	27	8	1	133	42	8	539
S06924	Frederick	50	> 30	Deicing2	7.4	158	49	11	1	139	69	9	665
S06925	Frederick	22	21 to 30	Deicing2	7.8	115	14	3	4	283	43	26	475
S06926	Frederick	0	0 to 5	Deicing2	7.5	112	4	3	7	141	15	7	623
S06927	Frederick	27	21 to 30	Deicing2	7.5	120	29	6	4	173	47	12	493
S06928	Frederick	0	0 to 5	Deicing2	7.5	118	4	3	1	93	13	6	545
S06929	Frederick	7	6 to 10	Deicing2	7.7	142	19	6	9	196	40	15	717
S06930	Frederick	26	21 to 30	Deicing2	8.1	131	18	2	4	273	49	28	292
S06931	Frederick	14	11 to 20	Deicing2	7.6	95	6	2	0	141	19	11	494
S06932	Frederick	0	0 to 5	Deicing2	7.5	303	5	5	0	106	16	7	681
S06933	Frederick	0	0 to 5	Deicing2	7.7	148	3	0	0	130	5	2	674
S06934	Frederick	21	21 to 30	Deicing2	8.0	79	8	1	3	64	13	4	413
S06935	Frederick	0	0 to 5	Deicing2	7.8	174	18	3	0	54	23	1	644
S06936	Frederick	41	> 30	Deicing2	7.9	126	35	2	1	180	55	18	455
S06937	Frederick	95	> 30	Deicing2	7.9	101	40	2	3	77	46	5	354
S06938	Frederick	10	6 to 10	Deicing2	7.9	154	28	2	4	185	47	17	667
S06939	Frederick	14	11 to 20	Deicing2	7.9	98	6	2	1	73	14	5	478
S06940	Frederick	30	> 30	Deicing2	7.7	186	39	13	12	216	64	12	938
S06941	Frederick	41	> 30	Deicing2	7.0	169	55	8	3	132	70	8	476
S06942	Frederick	6	6 to 10	Deicing2	7.3	160	2	11	3	264	34	22	689
S06943	Frederick	38	> 30	Deicing2	7.5	125	24	6	3	195	48	18	489
S06944	Frederick	0	0 to 5	Deicing2	7.5	60	0	1	1	65	4	3	119
S06945	Frederick	13	11 to 20	Deicing2	7.2	218	92	10	13	174	114	13	608

S06946	Frederick	17	11 to 20	Deicing2	7.5	105	20	7	2	152	39	12	324
S06947	Frederick	0	0 to 5	Deicing2	7.4	160	0	2	0	47	5	3	367
S06948	Frederick	25	21 to 30	Deicing2	7.4	108	15	6	5	159	33	12	468
S06949	Frederick	17	11 to 20	Deicing2	7.3	256	136	18	9	224	170	16	567
S06950	Frederick	52	> 30	Deicing2	7.7	58	18	1	1	73	26	6	104
S06951	Frederick	7	6 to 10	Deicing2	7.5	94	8	2	3	109	21	11	385
S06952	Frederick	19	11 to 20	Deicing2	7.4	132	40	11	2	140	58	7	423
S06953	Frederick	10	6 to 10	Deicing2	7.6	102	4	4	2	132	17	10	334
S06954	Frederick	7	6 to 10	Deicing2	7.5	156	30	11	6	161	53	12	651
S06955	Frederick	21	21 to 30	Deicing2	7.6	125	19	7	4	172	44	17	529

APPENDIX C

Table 18. Raw data for water extractable metals

NAWA			Age	Na	Ca	Mg	Na	K	B	S	Fe	Zn	Cu	Mn
									mg/kg					
S06887	Chicago	6	6 to 10	Deicing1	106	28	54	43	0	7	38	0	0	0
S06888	Chicago	21	21 to 30	Deicing1	141	38	120	82	0	17	28	1	0	0
S06889	Chicago	30	> 30	Deicing1	66	60	286	68	1	14	186	2	0	0
S06890	Chicago	7	6 to 10	Deicing1	149	31	112	59	0	25	16	0	0	0
S06891	Chicago	91	> 30	Deicing1	55	44	174	64	0	12	148	2	0	1
S06892	Chicago	72	> 30	Deicing1	58	64	320	96	1	13	239	3	0	1
S06893	Chicago	10	6 to 10	Deicing1	142	62	384	11	1	20	219	1	1	1
S06894	Chicago	0	0 to 5	Deicing1	171	36	95	56	0	11	110	1	1	1
S06895	Chicago	24	21 to 30	Deicing1	106	43	162	89	0	17	61	1	0	0
S06896	Chicago	14	11 to 20	Deicing1	104	40	115	52	1	14	137	1	0	0
S06897	Chicago	61	> 30	Deicing1	102	46	89	102	0	10	20	1	1	0
S06899	Chicago	92	> 30	Deicing1	183	49	158	76	0	15	19	1	0	0
S06900	Chicago	3	0 to 5	Deicing1	120	43	145	66	0	17	105	1	0	0
S06901	Chicago	11	11 to 20	Deicing1	176	42	27	79	0	11	105	1	0	0
S06902	Chicago	0	0 to 5	Deicing1	146	43	155	46	0	24	45	0	0	0
S06903	Chicago	3	0 to 5	Deicing1	213	53	16	47	0	19	28	1	0	0
S06904	Chicago	21	21 to 30	Deicing1	150	56	140	115	1	20	103	1	0	0
S06905	Chicago	12	11 to 20	Deicing1	89	49	310	118	0	14	129	1	0	0
S06906	Chicago	0	0 to 5	Deicing1	172	37	99	57	0	15	29	1	0	0
S06907	Chicago	7	6 to 10	Deicing1	74	60	201	82	1	12	229	1	0	1
S06908	Chicago	27	21 to 30	Deicing1	135	175	442	253	3	20	964	4	1	2
S06909	Chicago	12	11 to 20	Deicing1	90	43	387	183	1	30	190	2	1	1
S06910	Chicago	0	0 to 5	Deicing1	153	54	69	61	0	14	73	1	0	0
S06911	Chicago	18	11 to 20	Deicing1	86	71	258	133	1	11	326	2	0	2
S06912	Chicago	59	> 30	Deicing1	57	52	172	73	1	12	173	2	0	0
S06913	Chicago	55	> 30	Deicing1	93	79	396	128	1	18	320	2	0	1
S06914	Chicago	69	> 30	Deicing1	69	56	265	80	1	14	196	1	0	1
S06915	Chicago	9	6 to 10	Deicing1	155	47	19	48	0	11	49	1	0	0

S06916	Chicago	0	0 to 5	Deicing1	166	51	34	99	0	20	54	0	0	0
S06917	Chicago	8	6 to 10	Deicing1	118	47	179	53	0	11	126	1	0	1
S06918	Chicago	15	11 to 20	Deicing1	179	49	51	45	0	12	45	0	0	0
S06919	Chicago	10	6 to 10	Deicing1	200	41	20	48	0	16	39	1	0	0
S06920	Chicago	29	21 to 30	Deicing1	82	89	494	133	1	19	419	2	0	1
S06956	BCS	13	11 to 20	Irrigation	183	158	447	78	1	36	962	3	0	1
S06957	BCS	13	11 to 20	Irrigation	155	116	478	72	1	29	711	2	0	1
S06958	BCS	60	> 30	Irrigation	177	31	57	24	0	11	123	1	0	0
S06959	BCS	56	> 30	Irrigation	148	31	31	24	0	10	67	2	0	0
S06960	BCS	30	> 30	Irrigation	237	251	734	159	3	32	1501	4	2	3
S06961	BCS	0	0 to 5	Irrigation	103	28	140	36	0	11	96	1	0	0
S06962	BCS	28	21 to 30	Irrigation	109	56	322	52	1	12	451	2	1	1
S06963	BCS	60	> 30	Irrigation	44	24	74	33	0	9	107	2	1	0
S06964	BCS	8	6 to 10	Irrigation	173	23	122	39	0	18	54	1	1	0
S06965	BCS	13	11 to 20	Irrigation	454	185	1175	318	2	44	1900	3	1	2
S06966	BCS	9	6 to 10	Irrigation	146	49	237	76	0	20	159	1	0	0
S06967	BCS	0	0 to 5	Irrigation	138	28	140	36	0	26	20	1	0	0
S06968	BCS	34	> 30	Irrigation	104	17	10	47	0	6	47	1	0	0
S06969	BCS	9	6 to 10	Irrigation	130	75	324	57	0	51	337	1	0	1
S06970	BCS	28	21 to 30	Irrigation	90	29	228	60	0	15	144	1	0	1
S06971	BCS	0	0 to 5	Irrigation	109	41	145	122	0	20	177	1	0	0
S06972	BCS	27	21 to 30	Irrigation	93	20	322	55	0	19	96	0	0	0
S06973	BCS	0	0 to 5	Irrigation	179	32	179	51	0	131	58	1	0	0
S06974	BCS	23	21 to 30	Irrigation	176	156	705	135	2	52	1328	5	1	3
S06975	BCS	0	0 to 5	Irrigation	168	31	333	27	0	75	91	1	0	0
S06976	BCS	22	21 to 30	Irrigation	102	62	383	55	1	22	309	1	1	1
S06977	BCS	15	11 to 20	Irrigation	110	79	392	62	1	26	381	2	1	1
S06978	BCS	8	6 to 10	Irrigation	608	53	85	55	0	486	0	0	0	0
S06979	BCS	8	6 to 10	Irrigation	166	29	21	71	0	17	91	1	0	0
S06980	BCS	19	11 to 20	Irrigation	108	32	151	57	0	29	113	1	0	0
S06981	BCS	8	6 to 10	Irrigation	312	352	766	213	4	49	3478	5	1	4
S06982	BCS	34	> 30	Irrigation	280	201	832	90	3	38	1894	6	1	5
S06983	BCS	23	21 to 30	Irrigation	207	217	885	195	3	113	2414	5	1	5

S06984	BCS	11	11 to 20	Irrigation	73	33	89	45	0	9	130	1	0	0
S06985	BCS	34	> 30	Irrigation	136	40	407	32	1	31	171	1	0	0
S06986	BCS	9	6 to 10	Irrigation	149	123	548	68	1	34	491	2	0	2
S06987	BCS	0	0 to 5	Irrigation	127	33	186	32	0	23	103	1	0	0
S06988	BCS	27	21 to 30	Irrigation	207	236	320	202	3	39	4091	6	0	3
S06989	BCS	16	11 to 20	Irrigation	119	29	62	46	0	8	85	1	0	0
S06990	Galveston	30	> 30	Seasalt	112	23	20	62	0	9	46	2	0	0
S06991	Galveston	3	0 to 5	Seasalt	145	33	23	55	0	12	75	1	0	0
S06992	Galveston	7	6 to 10	Seasalt	138	31	44	57	0	13	33	1	0	0
S06993	Galveston	55	> 30	Seasalt	152	34	25	89	0	13	52	1	0	0
S06994	Galveston	50	> 30	Seasalt	145	32	32	62	0	11	66	1	0	0
S06995	Galveston	45	> 30	Seasalt	185	30	37	117	0	15	42	1	0	0
S06996	Galveston	6	6 to 10	Seasalt	147	23	26	62	0	12	54	1	0	0
S06997	Galveston	0	0 to 5	Seasalt	105	24	31	47	0	3	50	1	0	0
S06998	Galveston	7	6 to 10	Seasalt	41	3	8	10	0	3	6	0	0	0
S06999	Galveston	89	> 30	Seasalt	121	26	107	1394	1	19	20	1	0	0
S07000	Galveston	27	21 to 30	Seasalt	169	32	37	57	0	14	48	1	0	0
S07001	Galveston	12	11 to 20	Seasalt	148	36	14	59	0	9	90	1	0	0
S07002	Galveston	22	21 to 30	Seasalt	181	37	36	69	0	16	74	1	0	0
S07003	Galveston	2	0 to 5	Seasalt	152	30	24	70	0	14	84	1	0	0
S07004	Galveston	34	> 30	Seasalt	122	24	21	49	0	9	40	1	0	0
S07005	Galveston	15	11 to 20	Seasalt	156	24	11	47	0	8	59	1	0	0
S07006	Galveston	10	6 to 10	Seasalt	127	28	11	55	0	7	85	1	0	0
S07007	Galveston	18	11 to 20	Seasalt	158	31	24	56	0	11	54	1	0	0
S07008	Galveston	13	11 to 20	Seasalt	111	34	31	50	0	10	66	1	0	0
S07009	Galveston	25	21 to 30	Seasalt	112	28	22	49	0	8	61	1	0	0
S07010	Galveston	11	11 to 20	Seasalt	91	22	17	28	0	7	60	1	1	0
S07011	Galveston	28	21 to 30	Seasalt	99	24	22	26	0	8	44	1	0	0
S07012	Galveston	16	11 to 20	Seasalt	117	28	48	29	0	15	48	1	0	0
S07013	Galveston	5	0 to 5	Seasalt	80	18	19	37	0	6	33	1	0	0
S07014	Galveston	23	21 to 30	Seasalt	112	28	16	41	0	8	85	1	0	0
S07015	Galveston	10	6 to 10	Seasalt	128	26	47	47	0	19	49	0	0	0
S07016	Galveston	8	6 to 10	Seasalt	95	23	18	46	0	6	72	1	0	0

S07017	Galveston	21	21 to 30	Seasalt	79	16	22	66	0	5	36	1	0	0
S07018	Galveston	4	0 to 5	Seasalt	79	30	89	54	0	12	67	1	0	0
S07019	Galveston	35	> 30	Seasalt	111	25	30	43	0	6	65	1	0	0
S07020	Galveston	31	> 30	Seasalt	58	7	12	15	0	3	13	1	0	0
S07021	Galveston	14	11 to 20	Seasalt	36	9	23	14	0	6	29	1	0	0
S07022	Galveston	0	0 to 5	Seasalt	69	43	202	54	0	19	118	1	0	0
S07023	Galveston	9	6 to 10	Seasalt	115	24	19	52	0	20	45	1	0	0
S07024	Galveston	0	0 to 5	Seasalt	38	6	9	17	0	3	12	1	0	0
S06921	Frederick	26	21 to 30	Deicing2	190	8	28	8667	0	14	33	1	0	0
S06922	Frederick	7	6 to 10	Deicing2	194	10	16	125	0	11	62	0	0	0
S06923	Frederick	20	11 to 20	Deicing2	238	24	24	56	0	15	49	0	0	0
S06924	Frederick	50	> 30	Deicing2	193	9	169	38	0	14	30	1	0	0
S06925	Frederick	22	21 to 30	Deicing2	50	5	301	11	0	17	46	1	0	0
S06926	Frederick	0	0 to 5	Deicing2	206	18	15	97	0	14	15	1	0	0
S06927	Frederick	27	21 to 30	Deicing2	221	12	19	44	0	15	12	1	0	0
S06928	Frederick	0	0 to 5	Deicing2	227	15	10	45	0	29	1	0	0	0
S06929	Frederick	7	6 to 10	Deicing2	139	18	186	67	0	12	30	1	0	0
S06930	Frederick	26	21 to 30	Deicing2	28	14	339	30	0	18	162	1	0	1
S06931	Frederick	14	11 to 20	Deicing2	198	11	27	18	0	13	36	1	0	0
S06932	Frederick	0	0 to 5	Deicing2	556	38	47	83	0	312	0	1	0	0
S06933	Frederick	0	0 to 5	Deicing2	281	16	25	54	0	48	1	1	0	0
S06934	Frederick	21	21 to 30	Deicing2	101	7	109	11	0	8	8	1	0	0
S06935	Frederick	0	0 to 5	Deicing2	303	24	40	43	0	72	0	1	0	0
S06936	Frederick	41	> 30	Deicing2	141	9	171	14	0	13	41	1	0	0
S06937	Frederick	95	> 30	Deicing2	130	7	103	15	0	10	10	1	0	0
S06938	Frederick	10	6 to 10	Deicing2	145	10	235	42	0	22	62	1	0	0
S06939	Frederick	14	11 to 20	Deicing2	194	11	12	35	0	9	29	1	0	0
S06940	Frederick	30	> 30	Deicing2	318	35	11	99	0	16	15	1	0	0
S06941	Frederick	41	> 30	Deicing2	276	9	27	20	0	17	11	1	0	0
S06942	Frederick	6	6 to 10	Deicing2	228	26	23	99	0	15	48	1	0	0
S06943	Frederick	38	> 30	Deicing2	94	6	222	16	0	13	33	1	0	0
S06944	Frederick	0	0 to 5	Deicing2	79	22	17	20	0	28	28	1	0	1
S06945	Frederick	13	11 to 20	Deicing2	225	42	36	237	0	14	12	1	0	0

S06946	Frederick	17	11 to 20	Deicing2	127	9	90	28	0	14	26	1	0	0
S06947	Frederick	0	0 to 5	Deicing2	263	17	34	50	0	121	0	0	0	0
S06948	Frederick	25	21 to 30	Deicing2	175	10	47	70	0	17	22	0	0	0
S06949	Frederick	17	11 to 20	Deicing2	392	52	41	65	0	26	18	1	0	0
S06950	Frederick	52	> 30	Deicing2	50	7	77	18	0	6	63	1	0	1
S06951	Frederick	7	6 to 10	Deicing2	104	15	102	42	0	7	74	1	0	0
S06952	Frederick	19	11 to 20	Deicing2	207	22	22	56	0	11	66	1	0	0
S06953	Frederick	10	6 to 10	Deicing2	158	6	81	13	0	8	18	0	0	0
S06954	Frederick	7	6 to 10	Deicing2	264	24	15	89	0	15	19	1	0	1
S06955	Frederick	21	21 to 30	Deicing2	220	20	22	47	0	11	26	1	0	1

APPENDIX D

Table 19: Raw data for 2 hour shaken water extracts

NAWA ID	City	Age	Age Class	Na Source	pH	EC µS/cm	NO_3-N	NH_4-N	PO_4-P mg/kg	DOC	TDN	DON
S06887	Chicago	6	6 to 10	Deicing 1	7.94	107	8.2	12.3	0.9	156.7	13.5	0.0
S06888	Chicago	21	21 to 30	Deicing 1	8.04	140	47.1	13.9	3.7	221.7	63.2	2.1
S06889	Chicago	30	21 to 30	Deicing 1	8.47	112	13.7	3.5	4.6	375.2	48.8	31.7
S06890	Chicago	7	6 to 10	Deicing 1	7.98	106	17.4	4.2	3.5	186.8	33.5	11.9
S06891	Chicago	91	> 30	Deicing 1	8.4	64	22.5	3.7	5.5	179.3	37.1	10.9
S06892	Chicago	72	> 30	Deicing 1	8.38	80	22.4	3.5	3.8	185.1	36.7	10.8
S06893	Chicago	10	6 to 10	Deicing 1	8.6	192	14.8	5.4	3.9	474.8	50.7	30.5
S06894	Chicago	0	0 to 5	Deicing 1	8.32	122	4.4	15.6	0.7	139.6	10.1	0.0
S06895	Chicago	24	21 to 30	Deicing 1	8.61	122	6.8	5.8	4.2	276.8	28.4	15.8
S06896	Chicago	14	11 to 20	Deicing 1	8.3	80	8.9	3.7	2.5	282.4	30.0	17.4
S06897	Chicago	61	> 30	Deicing 1	8.18	66	20.6	1.6	2.7	168.7	34.3	12.1
S06898	Chicago	226	> 30	Deicing 1	8.32	180	30.8	3.7	5.9	136.8	41.0	6.6
S06899	Chicago	92	> 30	Deicing 1	8.17	44	19.6	0.8	2.6	73.2	21.3	1.0
S06900	Chicago	3	0 to 5	Deicing 1	8.18	89	9.9	1.1	2.2	141.1	19.8	8.8
S06901	Chicago	11	11 to 20	Deicing 1	8.18	96	13.2	0.9	1.6	107.5	19.0	4.8
S06902	Chicago	0	0 to 5	Deicing 1	8.89	107	6.8	0.8	0.2	138.1	13.3	5.6
S06903	Chicago	3	0 to 5	Deicing 1	8.15	125	26.7	2.0	2.0	123.2	35.9	7.3
S06904	Chicago	21	21 to 30	Deicing 1	8.36	156	16.1	3.1	17.5	260.4	33.7	14.5
S06905	Chicago	12	11 to 20	Deicing 1	8.79	179	23.5	1.9	6.6	296.2	58.5	33.1
S06906	Chicago	0	0 to 5	Deicing 1	6.51	115	2.0	1.2	0.4	130.9	11.6	8.4
S06907	Chicago	7	6 to 10	Deicing 1	8.43	94	15.6	1.0	6.0	208.0	36.2	19.6

S06908	Chicago	27	21 to 30	Deicing 1	9.03	178	6.4	2.7	6.4	554.2	58.5	49.5
S06909	Chicago	12	11 to 20	Deicing 1	8.88	145	6.8	1.2	11.0	265.5	34.1	26.1
S06910	Chicago	0	0 to 5	Deicing 1	8.42	70	5.3	0.6	2.9	133.8	18.2	12.4
S06911	Chicago	18	11 to 20	Deicing 1	8.62	105	30.8	2.1	8.3	292.3	61.4	28.5
S06912	Chicago	59	> 30	Deicing 1	8.64	87	11.2	1.2	6.8	252.2	37.2	24.8
S06913	Chicago	55	> 30	Deicing 1	8.59	128	6.8	1.5	7.5	520.1	51.1	42.8
S06914	Chicago	69	> 30	Deicing 1	8.52	94	12.4	1.4	7.8	339.4	45.7	31.9
S06915	Chicago	9	6 to 10	Deicing 1	7.96	62	9.7	1.3	2.2	191.2	30.0	18.9
S06916	Chicago	0	0 to 5	Deicing 1	7.92	103	14.5	1.3	3.2	134.7	28.8	13.0
S06917	Chicago	8	6 to 10	Deicing 1	8.2	96	7.3	1.9	4.2	275.6	39.7	30.4
S06918	Chicago	15	11 to 20	Deicing 1	8.19	136	6.9	1.0	1.9	188.8	26.4	18.5
S06919	Chicago	10	6 to 10	Deicing 1	8.18	87	9.4	2.7	4.2	128.1	23.9	11.7
S06920	Chicago	29	21 to 30	Deicing 1	8.93	178	18.9	3.0	8.9	387.8	62.1	40.2
S06921	Maryland	26	21 to 30	Deicing 2	7.32	77	5.1	4.9	1.1	130.2	22.0	12.0
S06922	Maryland	7	6 to 10	Deicing 2	8.26	95	6.2	2.3	1.2	174.9	19.6	11.0
S06923	Maryland	20	11 to 20	Deicing 2	6.8	108	23.9	3.8	1.4	159.0	41.2	13.5
S06924	Maryland	50	> 30	Deicing 2	7.52	143	45.4	9.1	2.0	148.1	65.9	11.4
S06925	Maryland	22	21 to 30	Deicing 2	8.43	97	11.1	2.0	3.7	239.2	36.8	23.6
S06926	Maryland	0	0 to 5	Deicing 2	7.9	106	5.4	1.8	4.2	142.7	14.2	7.0
S06927	Maryland	27	21 to 30	Deicing 2	7.68	97	24.6	3.8	3.6	131.4	36.5	8.0
S06928	Maryland	0	0 to 5	Deicing 2	7.39	120	5.6	2.6	0.9	124.7	19.7	11.5
S06929	Maryland	7	6 to 10	Deicing 2	7.81	105	13.3	4.3	8.2	169.5	33.3	15.8
S06930	Maryland	26	21 to 30	Deicing 2	8.77	105	16.3	1.5	2.2	203.9	39.9	22.1
S06931	Maryland	14	11 to 20	Deicing 2	7.64	70	4.4	4.3	0.4	138.5	17.8	9.0
S06932	Maryland	0	0 to 5	Deicing 2	7.7	292	3.7	5.1	0.4	124.0	13.5	4.7
S06933	Maryland	0	0 to 5	Deicing 2	7.66	127	3.9	1.5	0.4	110.3	6.7	1.3
S06934	Maryland	21	21 to 30	Deicing 2	7.71	86	9.8	1.9	1.9	84.5	15.8	4.1
S06935	Maryland	0	0 to 5	Deicing 2	7.86	177	19.0	5.1	0.4	70.5	24.3	0.1

S06936	Maryland	41	>30	Deicing 2	7.92	94	33.1	4.4	1.1	127.8	47.2	9.7
S06937	Maryland	95	>30	Deicing 2	7.81	90	41.9	2.6	2.1	74.6	48.7	4.2
S06938	Maryland	10	6 to 10	Deicing 2	8	145	25.5	3.4	3.3	202.9	50.3	21.3
S06939	Maryland	14	11 to 20	Deicing 2	7.47	93	8.4	2.2	0.7	67.0	11.6	1.0
S06940	Maryland	30	>30	Deicing 2	7.55	140	25.8	8.3	9.6	212.3	53.2	19.0
S06941	Maryland	41	>30	Deicing 2	7.18	115	52.6	4.6	2.1	123.9	62.6	5.4
S06942	Maryland	6	6 to 10	Deicing 2	7.47	127	4.7	5.8	1.3	163.2	20.1	9.5
S06943	Maryland	38	>30	Deicing 2	7.97	93	25.0	3.6	2.2	221.9	50.9	22.3
S06944	Maryland	0	0 to 5	Deicing 2	8.24	55	3.8	0.1	0.4	74.0	2.5	0.0
S06945	Maryland	13	11 to 20	Deicing 2	7.33	186	73.1	6.4	10.1	126.7	90.0	10.4
S06946	Maryland	17	11 to 20	Deicing 2	7.77	79	19.8	6.7	1.4	199.4	38.8	12.2
S06947	Maryland	0	0 to 5	Deicing 2	7.73	150	3.9	1.7	0.6	47.0	3.7	0.0
S06948	Maryland	25	21 to 30	Deicing 2	7.46	90	18.7	2.6	4.0	117.2	28.8	7.4
S06949	Maryland	17	11 to 20	Deicing 2	7.42	245	132.7	10.5	7.6	225.5	156.8	13.6
S06950	Maryland	52	>30	Deicing 2	8.1	35	17.2	3.8	0.5	82.8	24.2	3.1
S06951	Maryland	7	6 to 10	Deicing 2	8.09	86	9.7	2.9	1.2	117.4	18.2	5.7
S06952	Maryland	19	11 to 20	Deicing 2	7.64	106	41.1	8.0	1.0	161.1	59.2	10.1
S06953	Maryland	10	6 to 10	Deicing 2	7.78	93	4.7	1.5	1.0	114.6	17.9	11.7
S06954	Maryland	7	6 to 10	Deicing 2	7.81	138	29.1	6.4	5.0	140.8	49.7	14.2
S06955	Maryland	21	21 to 30	Deicing 2	7.56	86	15.9	5.3	2.1	171.0	36.5	15.3
S06990	Galveston	30	21 to 30	Seasalt	6.28	66	7.5	3.7	1.05	170.1	23.8	12.6
S06991	Galveston	3	0 to 5	Seasalt	7.5	60	5.0	1.3	1.14	188.2	24.1	0.0
S06992	Galveston	7	6 to 10	Seasalt	7.59	70	5.6	3.8	5.63	178.9	23.8	14.4
S06993	Galveston	55	> 30	Seasalt	7.61	69	5.4	3.5	2.73	177.2	22.8	13.9
S06994	Galveston	50	> 30	Seasalt	7.77	71	10.2	3.8	1.53	225.5	33.6	19.5
S06995	Galveston	45	> 30	Seasalt	7.87	90	10.5	4.4	3.00	205.6	31.7	16.9
S06996	Galveston	6	6 to 10	Seasalt	8.04	69	6.1	3.5	3.14	156.6	20.9	11.3
S06997	Galveston	0	0 to 5	Seasalt	8.12	61	16.2	4.8	3.23	98.6	27.1	6.1

S06998	Galveston	7	6 to 10	Seasalt	8.15	38	2.0	3.0	0.74	111.0	12.8	7.9
S06999	Galveston	89	> 30	Seasalt	8.14	68	5.4	5.6	7.65	305.2	37.0	26.0
S07000	Galveston	27	21 to 30	Seasalt	8.13	79	6.6	0.4	8.05	352.9	26.7	19.7
S07001	Galveston	12	11 to 20	Seasalt	8.05	58	7.2	0.2	2.14	148.8	22.0	14.7
S07002	Galveston	22	21 to 30	Seasalt	8.18	9	5.6	0.7	1.88	202.3	25.8	19.5
S07003	Galveston	2	0 to 5	Seasalt	8.29	64	5.6	0.5	1.93	130.3	19.4	13.4
S07004	Galveston	34	> 30	Seasalt	8.04	49	4.6	0.4	5.29	184.8	22.1	17.1
S07005	Galveston	15	11 to 20	Seasalt	8	56	6.5	3.6	3.10	162.8	22.7	12.6
S07006	Galveston	10	6 to 10	Seasalt	8.18	55	3.9	0.8	3.66	135.2	19.5	14.7
S07007	Galveston	18	11 to 20	Seasalt	8.51	73	6.0	0.0	2.39	200.3	25.0	18.9
S07008	Galveston	13	11 to 20	Seasalt	8.41	62	4.5	0.4	0.98	123.1	18.2	13.2
S07009	Galveston	25	21 to 30	Seasalt	8.76	59	3.6	3.3	1.26	96.3	13.5	6.6
S07010	Galveston	11	11 to 20	Seasalt	8.48	40	3.2	0.2	0.68	83.9	12.2	8.8
S07011	Galveston	28	21 to 30	Seasalt	8.4	59	5.1	0.4	1.31	136.8	19.4	14.0
S07012	Galveston	16	11 to 20	Seasalt	8.54	69	3.8	1.0	2.64	143.7	21.3	16.5
S07013	Galveston	5	0 to 5	Seasalt	8.45	40	4.8	0.7	3.13	108.7	16.4	10.9
S07014	Galveston	23	21 to 30	Seasalt	8.38	55	2.7	0.0	0.87	101.6	13.8	11.1
S07015	Galveston	10	6 to 10	Seasalt	8.86	90	11.5	0.0	0.44	85.4	20.2	8.7
S07016	Galveston	8	6 to 10	Seasalt	8.97	44	2.1	0.0	0.72	79.2	8.9	6.7
S07017	Galveston	21	21 to 30	Seasalt	8.85	39	4.0	0.1	1.47	80.8	13.1	9.0
S07018	Galveston	4	0 to 5	Seasalt	9.18	73	0.8	0.1	1.12	89.4	9.5	8.6
S07019	Galveston	35	> 30	Seasalt	8.91	54	4.2	0.7	3.28	94.8	13.5	8.5
S07020	Galveston	31	> 30	Seasalt	8.86	48	5.6	0.4	1.35	88.4	15.8	9.9
S07021	Galveston	14	11 to 20	Seasalt	8.03	24	6.9	0.0	0.88	100.7	17.6	10.7
S07022	Galveston	0	0 to 5	Seasalt	9.05	118	1.2	0.8	0.66	89.5	9.0	6.9
S07023	Galveston	9	6 to 10	Seasalt	9.18	69	3.8	0.7	1.75	130.2	18.7	14.1
S07024	Galveston	0	0 to 5	Seasalt	9.09	43	3.4	0.0	0.99	77.4	11.5	8.0
S06956	BCS	13	11 to 20	Irrigation	8.96	198	8.1	6.9	2.49	363.5	33.6	18.6

S06957	BCS	13	11 to 20	Irrigation	9.57	201	7.6	3.7	2.66	322.8	29.9	18.6
S06958	BCS	60	>30	Irrigation	8.37	70	9.9	3.0	1.70	233.6	27.0	14.0
S06959	BCS	56	>30	Irrigation	8.01	55	8.9	0.7	3.17	251.8	25.9	16.3
S06960	BCS	30	>30	Irrigation	9.85	298	10.4	3.1	6.06	709.3	75.5	61.9
S06961	BCS	0	0 to 5	Irrigation	9.05	89	5.7	0.6	2.34	183.7	15.5	9.2
S06962	BCS	28	21 to 30	Irrigation	9.36	134	8.8	9.5	3.76	268.2	34.2	15.9
S06963	BCS	60	>30	Irrigation	9.26	36	7.0	1.2	11.42	251.5	27.3	19.2
S06964	BCS	8	6 to 10	Irrigation	8.46	121	6.0	4.5	1.06	200.6	19.1	8.7
S06965	BCS	13	11 to 20	Irrigation	9.89	428	12.3	3.8	6.39	791.7	85.2	69.1
S06966	BCS	9	6 to 10	Irrigation	8.94	147	7.3	1.6	7.98	313.7	35.6	26.6
S06967	BCS	0	0 to 5	Irrigation	8.57	82	5.8	0.9	1.42	120.4	12.5	5.8
S06968	BCS	34	>30	Irrigation	8.12	42	6.6	1.1	2.16	174.4	14.3	6.6
S06969	BCS	9	6 to 10	Irrigation	8.83	164	6.4	2.1	4.05	279.0	29.2	20.6
S06970	BCS	28	21 to 30	Irrigation	8.92	131	9.4	2.8	4.79	239.7	31.9	19.7
S06971	BCS	0	0 to 5	Irrigation	8.76	110	5.7	1.6	1.90	94.4	4.8	0.0
S06972	BCS	27	21 to 30	Irrigation	8.67	172	6.7	3.0	2.67	376.4	35.4	25.7
S06973	BCS	0	0 to 5	Irrigation	8.37	212	4.2	0.5	0.67	151.2	4.2	0.0
S06974	BCS	23	21 to 30	Irrigation	9.78	327	9.5	1.7	5.81	453.1	55.0	43.7
S06975	BCS	0	0 to 5	Irrigation	9.15	204	4.4	0.9	0.73	173.6	12.5	7.2
S06976	BCS	22	21 to 30	Irrigation	9.69	208	6.6	1.6	2.89	313.0	37.1	28.9
S06977	BCS	15	11 to 20	Irrigation	9.43	166	6.1	1.9	3.81	285.7	31.3	23.3
S06978	BCS	8	6 to 10	Irrigation	8.51	252	4.8	1.5	1.10	178.4	12.3	6.0
S06979	BCS	8	6 to 10	Irrigation	8.61	91	7.1	2.0	4.56	171.4	17.8	8.8
S06980	BCS	19	11 to 20	Irrigation	9.8	281	12.1	2.7	4.88	517.3	56.8	42.0
S06981	BCS	8	6 to 10	Irrigation	8.81	124	6.3	1.4	4.00	220.4	23.7	16.0
S06982	BCS	34	>30	Irrigation	9.93	368	10.1	2.8	6.92	819.5	87.3	74.4
S06983	BCS	23	21 to 30	Irrigation	9.88	411	10.3	3.2	4.74	552.5	64.9	51.4
S06984	BCS	11	11 to 20	Irrigation	8.69	53	6.4	0.8	3.08	217.0	16.2	9.0

S06985	BCS	34	>30	Irrigation	9.51	226	5.0	1.8	1.34	84.1	4.4	0.0
S06986	BCS	9	6 to 10	Irrigation	9.69	255	6.0	2.4	3.96	291.4	31.0	22.6
S06987	BCS	0	0 to 5	Irrigation	8.94	128	4.4	0.5	1.04	141.8	6.4	1.5
S06988	BCS	27	21 to 30	Irrigation	8.8	113	10.4	5.6	3.49	651.9	59.0	43.0
S06989	BCS	16	11 to 20	Irrigation	8.45	77	4.9	1.1	1.40	215.3	22.5	16.5

APPENDIX E

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